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BULLETIN NO. 56.

U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

PROCEEDINGS

OF THE

FIFTEENTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

HELD AT

WASHINGTON, D. C.,

NOVEMBER 11, 12, AND 14, 1898.

EDITED BY

HARVEY W. WILEY,

SECRETARY OF THE ASSOCIATION.

WASHINGTON:

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LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,
Washington, D. C., January 17, 1899.

SIR: I have the honor to transmit to you herewith the manuscript of the proceedings of the fifteenth annual convention of the Association of Official Agricultural Chemists, with the request that it be published as Bulletin No. 56 of the Division of Chemistry.

H. W. WILEY,
Chief of the Division of Chemistry and Secretary of the Association of Official Agricultural Chemists.

HON. JAMES WILSON,
Secretary of Agriculture.

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PROCEEDINGS OF THE FIFTEENTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

FIRST DAY.

FRIDAY—MORNING SESSION.

The fifteenth annual convention of the Association of Official Agricultural Chemists, beginning November 11, 1898, was called to order in the lecture hall of the Columbian University, by the president, Mr. A. L. Winton, at 10.15 a. m.

MEMBERS AND VISITORS PRESENT.

The following members and visitors were in attendance:

Allen, E. W., Office of Experiment Stations, Department of Agriculture, Washington, D. C.
Alvord, H. E., Dairy Division, Department of Agriculture, Washington, D. C.
Anderson, Cyrus N., Washington, D. C.
Andrews, W. H., Agricultural Experiment Station, Geneva, N. Y.
Atwater, W. O., Storrs Experiment Station, Middletown, Conn.
Bartlett, J. M., Agricultural Experiment Station, Orono, Me.
Beal, W. H., Office of Experiment Stations, Department of Agriculture, Washington, D. C.
Bigelow, W. D., Division of Chemistry, Department of Agriculture, Washington, D. C.
Blair, A. W., Agricultural Experiment Station, Raleigh, N. C.
Bolton, H. Carrington, Washington, D. C.
Cameron, F. K., Division of Soils, Department of Agriculture, Washington, D. C.
Cameron, George W., College Park, Md.
Carpenter, F. B., Virginia-Carolina Chemical Company, Richmond, Va.
Carr, Oma, Corry, Pa.
Coates, Charles E., Louisiana State University, Baton Rouge, La.
Craighill, George P., Lynchburg, Va.
Ewell, E. E., Division of Chemistry, Department of Agriculture, Washington, D. C.
Frear, Wm., Agricultural Experiment Station, State College, Pa.
Gaines, Richard H., Department of Agriculture, Richmond, Va.
Gascoyne, W. J., Baltimore, Md.
Goessmann, C. A., Hatch Experiment Station, Amherst, Mass.
Goessmann, C. I., Hatch Experiment Station, Amherst, Mass.
Grindley, H. S., Urbana, Ill.
Hartwell, B. L., Agricultural Experiment Station, Kingston, R. I.
Haywood, J. K., Division of Chemistry, Department of Agriculture, Washington, D. C.

Hills, J. K., Agricultural Experiment Station, Burlington, Vt.
Hiltner, R. S., Agricultural Experiment Station, Lincoln, Nebr.
Hite, B. H., Agricultural Experiment Station, Morgantown, W. Va.
Holland, E. B., Hatch Experiment Station, Amherst, Mass.
Huston, H. A., Agricultural Experiment Station, Lafayette, Ind.
Jaffa, M. E., Agricultural Experiment Station, Berkeley, Cal.
Jones, C. H., Agricultural Experiment Station, Burlington, Vt.
Kedzie, R. C., Agricultural College, Agricultural College, Mich.
Kerr, G. A., Columbus, Ind.
Kilgore, B. W., Agricultural and Mechanical College, College, Miss.
Krug, William H., Division of Chemistry, Department of Agriculture, Washington, D. C.
Langworthy, C. F., Office of Experiment Stations, Department of Agriculture, Washington, D. C.
Laughlin, J. R., Agricultural College, College Park, Md.
McCarty, R. Lee, New York.
McDonnell, H. B., Agricultural College, College Park, Md.
Meng, James S., New York.
Mitchell, W. L., Agricultural Experiment Station, New Haven, Conn.
Moore, C. C., Division of Chemistry, Department of Agriculture, Washington, D. C.
Morse, F. W., College of Agriculture and Mechanical Arts, Durham, N. H.
Myers, John A., New York.
Palmer, T. Chalkley, Philadelphia, Pa.
Patterson, H. J., Agricultural Experiment Station, College Park, Md.
Peebles, A. B., Storrs Experiment Station, Storrs, Conn.
Ross, B. B., Agricultural Experiment Station, Auburn, Ala.
Runyan, E. G., Division of Chemistry, Department of Agriculture, Washington, D. C.
Schulte, J. I., Washington, D. C.
Schweinitz, de, E. A., Bureau of Animal Industry, Department of Agriculture, Washington, D. C.
Scovell, M. A., Agricultural Experiment Station, Lexington, Ky.
Seal, Alfred N., Washington, D. C.
Skinner, W. W., Agricultural College, College Park, Md.
Snyder, Harry, Agricultural Experiment Station, St. Anthony Park, Minn.
Stewart, J. H., Agricultural Experiment Station, Morgantown, W. Va.
Stokes, H. N., Geological Survey, Washington, D. C.
Street, John Phillips, Agricultural Experiment Station, New Brunswick, N. J.
Trescot, T. C., Division of Chemistry, Department of Agriculture, Washington, D. C.
True, A. C., Office of Experiment Stations, Department of Agriculture, Washington, D. C.
Van Slyke, L. L., Agricultural Experiment Station, Geneva, N. Y.
Veitch, F. P., Agricultural College, College Park, Md.
Voorhees, L. A., Agricultural Experiment Station, New Brunswick, N. J.
Wheeler, H. J., Agricultural Experiment Station, Kingston, R. I.
Wiley, H. W., Division of Chemistry, Department of Agriculture, Washington, D. C.
Williams, C. B., Agricultural Experiment Station, Raleigh, N. C.
Winton, A. L., Agricultural Experiment Station, New Haven, Conn.
Withers, W. A., Agricultural Experiment Station, Raleigh, N. C.
Woll, F. W., Agricultural Experiment Station, Madison, Wis.
Woods, Charles D., Agricultural Experiment Station, Orono, Me.
Yocum, J. H., New York.

ORDER OF BUSINESS.

In compliance with the action taken at the last meeting of the association, the executive committee reported the following order of business:

The president's address.

Reports of the referees in the following order:

First. Report on nitrogen.

Second. Report on potash.

Third. Report on phosphoric acid.

Fourth. Report on soils and ash.

Fifth. Report on foods and feed stuffs.

Sixth. Report on food adulteration.

Seventh. Report on dairy products.

Eighth. Report on sugars.

Ninth. Report on tannin.

Tenth. Report on nicotin.

Eleventh. Reports of special committees (abstract committee, food standards, uniform laws for fertilizer control, volumetric standards, rewriting methods of soil analysis).

The president appointed Messrs. McDonnell, Bigelow, and Wheeler a committee on time of meeting. The committee recommended that two sessions be held daily, one from 9.30 a. m. to 12 noon and the second from 1.15 to 4 p. m. The recommendation of the committee was adopted.

The president made the special order for the election of officers for 2 p. m. on Saturday.

The president of the association then read the following annual address:

ADDRESS OF THE PRESIDENT.

Members of the Association of Official Chemists: Our association came into existence at a time when there was especial need of such an organization. The official agricultural laboratories in the United States were few in number, limited in experience and resources, and continually harassed by controversies with fertilizer manufacturers over the accuracy of their analytical work. The cooperative study of methods, which was undertaken soon after organization was effected, brought the individual chemists into closer touch, and the methods adopted not only came into use in all the official laboratories, but were soon recognized as standard methods by the trade.

The passage of the Hatch act gave to each State abundant means for agricultural investigation, a considerable part of which was devoted to chemical work. But as the laboratories increased in numbers and efficiency the demands on the association, instead of becoming less, became greater, and much discretion was required in determining the policy to be pursued. To-day, although we have been organized for

over fourteen years, we are still confronted by a number of questions of policy which demand our careful attention.

The first of these concerns the scope of the association. The name under which we are known gives little idea of our purpose and work. Agricultural chemistry, in the full significance of the term, is not merely a branch of chemistry, but is a comprehensive science which includes animal and plant physiology, physics, and other sciences so far as they are applied to agriculture. The well-known Jahresbericht über die Fortschritte auf dem Gesamtgebiete der Agrikultur Chemie for 1896 reviews the work of the year under the following heads: atmosphere, water, soils, fertilizers, physiology of plants, constituents of plants, seed testing, plant culture, plant diseases, fodders, constituents of animal bodies, chemico-physiological experiments on animals (including bees, silkworms, and fishes), animal nutrition, meat and milk production, dairy products, starch production, sugar production, diseases of the sugar beet, wine, liquors, and, finally, methods of analysis. Of the 700 pages but 55 are devoted to the latter subject.

Notwithstanding this long list of subjects which come within the scope of agricultural chemistry, we, as an association, have confined our attention almost entirely to matters pertaining to agricultural analysis. Our everyday work, it is true, is carried on in the field and in the stable as well as in the laboratory; but once a year we leave the crops and the farm animals behind and meet as analytical chemists.

During the first two years of its existence our organization was really a body of fertilizer analysts, as only methods for the analysis of these materials were considered; but at the third convention the constitution was amended so as to include as subjects for investigation "The analysis of soils, cattle foods, dairy products, and other materials connected with agricultural industry." The subjects fodders and dairy products were referred that year (1886) to committees, and sugar and distilled liquors in the following year. The first reporter on soils and ash was appointed in 1890 and on tannin in 1894. The methods for the analysis of dairy products and liquors which were tested included those for the detection of adulteration, and in 1896 this line of investigation was extended so as to cover all articles used for food and drink, a task which bids fair to become one of the most important we have yet undertaken.

Although from time to time the field of our labor has been enlarged, it really has never been extended beyond the bounds of chemical analysis, and it is believed that we have attained a much higher degree of usefulness under these limitations than would have been possible in a broader field.

But, while it is advisable that we remain an association of agricultural analysts, we should be this in the fullest sense. At the present convention Dr. de Schweinitz is expected to present a paper on the determination of nicotin in tobacco powders and tobacco extracts.

These products are coming into extensive use as insecticides and, as their value is dependent chiefly on the percentage of nicotin they contain, it is important that suitable methods be employed in determining this ingredient.

In addition to tobacco preparations, various other insecticides (chiefly arsenical powders), as well as fungicides, are frequently analyzed in official laboratories, and it seems to me desirable that a referee on insecticides and fungicides be appointed each year, to investigate methods for the determination of the valuable constituents of these products.

There is no immediate prospect that a further expansion of our work will be found advisable. The agricultural chemist may be called upon occasionally to analyze other classes of materials, such as, for example, well waters, paints for farm buildings, lubricants for farm machinery, and even wall paper for farm houses and grange halls, but these do not demand our attention as an association.

The second problem which confronts us is whether referees are to conduct cooperative investigations solely to study analytical methods, or, in part, to determine the accuracy of the work of the individual analysts. At the outstart samples were distributed, chiefly with the view of comparing the results obtained in different laboratories by the same methods. Such comparisons, while very instructive indeed to those making the analyses, did not, however, bring out any facts of general interest and it soon became apparent that results of more permanent value could be secured in the study of analytical methods. This latter work was accordingly taken up and has since resulted in many improvements of analytical processes, both as to accuracy and convenience. Unfortunately, the referees have been seriously hampered in their investigations, owing to the practice in some laboratories of using the samples sent out by the referees for a test of analysts rather than of methods. As a consequence of this abuse, it has been difficult to decide whether the method or the analyst was at fault. The referees are selected because of special qualifications for their work and they, in turn, should choose associates who are not only skilled manipulators, but have given particular attention to the subject under investigation. Furthermore, every official chemist should have it impressed upon him that it is better to decline to take part in an investigation than to assign the details to an inexperienced assistant.

Our plan of testing methods by the analysis of a few samples sent out by the referees, while in some respects an excellent one, is still open to grave criticism. If a considerable number of chemists secure good results by a new method in the analysis of three or four samples, the accuracy of the method is too often regarded as established. The fact is overlooked that it is the samples which have been thoroughly tested, whereas the method has been given only a preliminary trial. A method which works well on four samples but fails in the fifth is a snare and a

delusion. It is, therefore, of vastly greater importance that a wide variety of materials be examined by a few expert analysts than that a few materials be examined by a large number of analysts, and the best way to secure this variety is for each analyst to select his own materials. The analyst must, of course, make comparative determinations on each sample by the method under trial and a standard method, and for the purpose of eliminating from the comparison any possible errors due to variation in the sample, imperfect solution, or other cause, he should, whenever possible, use aliquots from the same solution. Many times these comparisons can be made on samples analyzed in connection with station work, and the results will have an immediate value, aside from throwing light on the methods.

The action taken at the last convention, leading to the appointment of a committee to consider standards of purity of the foods and drinks on sale in the United States, was but another manifestation of the growing determination to suppress adulteration and misbranding. The adoption of standards of purity should go hand in hand with the adoption of methods of examination, as the proper interpretation of results is as essential as the accuracy of their determination.

The undertaking is, however, fraught with difficulties and dangers and involves much patient and thoughtful work. Above all things we should avoid undue haste, as mistakes at the beginning will work incalculable harm.

In the case of some classes of foods, the data at hand are insufficient to serve as a suitable basis for standards and there is need of more analyses of authentic samples. This is particularly true of the spices, which subject was assigned to me by the committee. A considerable number of authentic samples, that is to say, samples ground in the laboratory from whole spices, were examined at the Department of Agriculture over eleven years ago, as described in Bulletin 13, part second, Spices and Condiments, but these results should be supplemented by analyses of the grades now on our markets. Some three months ago, I wrote to leading spice importers in New York City and Boston, asking if such samples could be secured. Three of the largest houses in New York and one in Boston at once replied that I was at liberty to sample all the goods in their warehouses from the original, unbroken packages. Through their courtesy over one hundred samples, representing all grades and qualities, have been drawn and the laborious task of analyzing these samples is now under way.

It thus appears that the work of fixing standards is not merely clerical, but involves the making of a large number of chemical analyses.

Another difficulty in fixing standards is the wide variation in quality, and, consequently, in composition, of foods. In our investigation of the spices this has already become apparent. For example, Singapore black pepper is a hard and perfect berry and, consequently, contains a higher percentage of starch and a lower percentage of fiber than the

lower grades of Acheen pepper, in which there are many empty shells. Ginger and cassia also vary greatly in quality and composition.

If we adopt standards based on analyses of the better grades, we shall be liable to condemn as adulterated inferior, but genuine, products, and thus work great injustice. It is, therefore, of the utmost importance that all grades be examined before attempting to fix standards.

Again, it is often difficult to decide as to what constitutes adulteration. A few examples drawn again from the spices will make this point clear. Black pepper, as you all know, is the unripe berry of the pepper plant, dried without shelling; white pepper is the mature berry from which the outer coatings have been removed. The pepper shells obtained as a by-product in preparing white pepper are extensively mixed with ground black pepper. This practice is regarded by food analysts as an adulteration analogous to the addition of skim milk to whole milk. The spice grinders, as a rule, take the ground that, since the pepper shells are a product from pepper which have about the same strength and chemical composition as the low grades of black pepper, their use is legitimate. They say that, even if it is granted, for the sake of argument, that pepper shells are an adulterant, it is impossible to distinguish between ground black pepper thus adulterated and that obtained by grinding a low-grade Acheen pepper, which contains shells as an accidental impurity.

The addition of ground clove stems to ground cloves is commonly prohibited, as the former contain but about 4 per cent of volatile oil, whereas the cloves contain upward of 20 per cent. The grinders claim that, since clove stems are always present in small amount in cloves as an accidental impurity, they should be rated as a low grade of cloves.

Assuming that both pepper shells and clove stems are adulterants, where shall the line be drawn between accident and design?

A similar problem was encountered years ago in fixing milk standards. It was evident that standards based on the minimum percentage of solids and fat which genuine milk ever contained would be so low as to be of little value in preventing adulteration. The only solution of the problem was to adopt arbitrary standards, so that milk which was abnormally low in nutritive value could not be sold under the name "milk," whether or not it had been skimmed or watered.

Another important question concerns the coloring of foods. Some are of the opinion that for the proper enforcement of pure-food laws it is necessary to regard all artificial coloring matters as adulterants, except when their presence is distinctly stated on the package, while others contend that only dyes injurious to health, or those added to conceal damage or the presence of makeweights, should be placed in this category. Whatever decisions are reached in this matter, common justice demands that the same rule shall be applied to one class of foods as to another. If the dairyman is allowed to color his butter and the

farmer his vinegar, the spice grinder should certainly have the privilege of adding to mustard enough turmeric to give it the attractive color which the housewife demands—a practice which a gentleman high in the trade has pronounced in my hearing as no more culpable than decorating the dining table with flowers or garnishing beefsteak with parsley.

These illustrations have been drawn from the spices for the reason that I am at present specially interested in these products; but similar difficulties are to be encountered in fixing standards for other classes of foods, and great care should be taken in reaching final decisions.

I have purposely omitted all mention of the specific problems in analytical chemistry for which we are seeking solutions, as these will be fully presented by the referees during our sessions. There is no limit to the work in this our chosen field. The methods for the proximate analyses of fodders and soils especially need improvement, and even the subjects nitrogen, phosphoric acid and potash, over which we have labored since our first convention, are not yet exhausted.

It is well that there are many workers and that there is a division of the labor.

APPOINTMENT OF COMMITTEES.

The president appointed the following committee on recommendations of referees: Messrs. Huston, Ross, and Street, together with the referee whose recommendations were to be considered.

The president appointed Messrs. Kedzie and Jaffa as a committee to wait upon the Secretary and Assistant Secretary of Agriculture and invite them to attend the meeting.

The preliminary business having been accomplished, the president announced that the order of business which had, by the vote of the previous convention, been left with the executive committee would now be followed, and called first for the reading of the report of the referee on nitrogen. The referee, Mr. R. J. Davidson, being absent on account of illness, his report was read by the secretary.

REPORT ON NITROGEN.

By R. J. Davidson, *Referee.*

In outlining the work for the year the referee tried to follow as closely as possible the instructions and suggestions made at the last meeting of the association.

It was recommended that a further investigation of the Ulsch-Kjeldahl and Ulsch-Gunning methods for total nitrogen be made, and in addition to continue the work on methods for the determination of available nitrogen in mixed fertilizers, with special reference to rapidity and reliability.

About the middle of February a letter was sent to a number of chemists asking them to take part in the work, and favorable responses were received from eleven. Some of these stated that they would do only part of the work. Eleven sets of materials were sent out about March 8, and about a month later another set was called for.

From these twelve sets of samples I have received up to the present time reports from two chemists on all the work, and from eight, reports on part of it.

For work in comparing the Ulsch-Kjeldahl and Ulsch-Gunning methods with the official methods, the following letter of instructions was sent:

MARCH 8, 1898.

DEAR SIR: With the organic sample I also send you two samples to be used in testing the Ulsch-Kjeldahl and Ulsch-Gunning methods for total nitrogen in sample rich in nitrate salts.

Sample No. 11 consists of a mixture of sodium nitrate, cottonseed meal, acid phosphate, and sodium chlorid.

Sample No. 12 consists of a mixture of sodium nitrate, cottonseed meal, tankage, and acid phosphate.

You are requested to make the following determinations in each sample:

A.—Total nitrogen by Kjeldahl and Gunning methods modified to include nitrates.

B.—Total nitrogen by Ulsch-Kjeldahl method.

C.—Total nitrogen by { Ulsch-Gunning method. / Ulsch-Kjeldahl method.

Place 1 gram of sample in a Kjeldahl flask, add 20 cc of water, shake, and allow to stand for ten minutes. Then add 3.5 grams of powdered iron (reduced iron answers very well) and 10 cc of sulphuric acid (sp. gr. 1.35), warm gently until evolution of hydrogen ceases; add 0.7 gram of mercuric oxid and 20 cc of strong sulphuric acid, and then proceed as in the regular Kjeldahl method.

ULSCH-GUNNING METHOD.

Proceed as in Ulsch-Kjeldahl method until conversion has been made with reduced iron and sulphuric acid, and then follow the regular Gunning method, using from 25 to 30 cc sulphuric acid for digestion instead of 20 cc.

It is requested that all results be reported as soon as possible.

Respectfully, yours,

R. J. DAVIDSON, *Referee.*
F. S. SHIVER, *Associate.*

The methods given above are the same as those used last year, except as to the quantity of reduced iron, which has been changed from 2 to 3.5 grams.

The materials used in the preparation of the above samples were as follows:

Sample No. 11:

20 grams nitrate of soda, containing 3.22 grams nitrogen.
100 grams cottonseed meal, containing 7.60 grams nitrogen.
100 grams acid phosphate.
70 grams sodium chlorid.

This sample contains 8.48 per cent of total nitrogen, of which 6.85 per cent are nitric nitrogen.

Sample No. 12:

150 grams nitrate of soda, containing 24.165 grams nitrogen.
100 grams cottonseed meal, containing 7.60 grams nitrogen.
100 grams tankage, containing 5.89 grams nitrogen.
170 grams acid phosphate.

This sample contains 7.24 per cent of total nitrogen, of which 4.65 per cent are nitric nitrogen.

In preparing these mixtures it was intended to have considerable nitric nitrogen present to test the serious objections raised at the last meeting against the Ulsch-Kjeldahl and Ulsch-Gunning methods.

It was stated then that the quantity of iron used (2 grams) was not sufficient when the per cent of nitric nitrogen present was 4 or over.

Before sending out these samples both the referee and the associate referee made several determinations, using 2, 2.5, 3, 3.5, and 4 grams of iron with 1 gram substance, to find out what was the smallest quantity of iron necessary, and it was found that not less than 3.5 grams should be used for No. 11, and that perhaps 4

grams would be better. But, as the main object of this investigation was to obtain information as to the amount of iron that should be used, it was thought best to limit the amount to 3.5 grams.

Comparison of methods.—Total nitrogen.

Analyst.	11					12				
	Modified Kjeldahl.	Modified Gunning.	Absolute.	Ulsch-Kjeldahl.	Ulsch-Gunning.	Modified Kjeldahl.	Modified Gunning.	Absolute.	Ulsch-Kjeldahl.	Ulsch-Gunning.
	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.
A. W. Ogden, Conn. Station	8.36	8.34	.....	8.42	8.34	7.21	7.21	.....	7.24	7.17
	8.38	8.36	.....	8.48	8.37	7.23	7.23	.....	7.27	7.18
	8.42	8.36	.....	.....	8.38	7.27	7.27	.....	7.28	7.20
	8.48	8.41	.....	.....	.....	7.28	7.30	.....	7.30	7.22
F. Thompson, N.Y. Station	.....	8.38	.....	.....	[1]8.37	.....	7.20	.....	.....	7.20
	.....	8.44	.....	.....	[1]8.41	.....	7.20	.....	.....	7.16
	.....	.....	.....	.....	.....	.....	7.31	.....	.....	7.18
T. C. Trescot, U. S. Dept. Agr	.....	[2]8.74	[2]8.72	.....	[2]8.70	.....	[2]7.46	[2]7.47	.....	[2]7.48
	.....	[2]8.76	[2]8.69	.....	[2]8.70	.....	[2]7.51	[2]7.41	.....	[2]7.45
	.....	[2]8.70	.....	.....	[2]8.76	.....	[2]7.51	.....	.....	.....
	.....	.....	.....	.....	.....	.....	[2]7.47	.....	.....	.....
Walker Bowman, Louisville Spirit-Cured Tobacco Co	8.57	.....	.....	.....	.....	7.26	.....	.....	.....	.....
C. H. Jones, Vt. Station	8.47	.....	.....	8.40	8.41	7.24	.....	.....	7.21	7.21
	8.48	.....	.....	8.39	8.40	7.25	.....	.....	7.19	7.27
	8.56	.....	.....	8.42	8.44	7.25	.....	.....	7.16	7.24
	8.54	.....	.....	.....	.....	7.19	.....	.....	.....	.....
	8.57	.....	.....	.....	.....	.....	.....	.....	.....	.....
H. K. Miller, N. C. Station	8.39	.....	.....	8.40	.....	7.32	.....	.....	7.20	.....
	8.36	.....	.....	8.30	.....	7.35	.....	.....	7.11	.....
	8.41	.....	.....	8.30	.....	.....	.....	.....	7.26	.....
	[2]8.27	.....	.....	.....	.....	.....	.....	.....	.....	.....
W. B. Ellett, Va. Station	8.56	.....	.....	8.34	.....	7.22	.....	.....	7.21	7.21
	8.48	.....	.....	8.38	8.39	7.35	.....	.....	7.20	7.26
A. T. Eskridge, Va. Station	.....	.....	.....	8.49	.....	.....	.....	.....	7.34	.....
	8.47	.....	.....	8.38	8.30	.....	.....	.....	7.21	.....
J. P. Street, N. J. Station	8.34	.....	.....	8.25	8.25	7.20	.....	.....	7.13	7.16
	8.34	.....	.....	8.31	8.25	7.21	.....	.....	7.19	7.13
	8.34	.....	.....	8.28	8.25	7.21	.....	.....	7.16	7.15
F. S. Shiver, S. C. Station	8.56	.....	.....	8.57	8.63	7.29	.....	.....	7.28	7.23
F. P. Veitch, Md. Station	.....	8.32	.....	.....	.....	.....	7.14	.....	.....	.....
	.....	8.36	.....	.....	.....	.....	7.16	.....	.....	.....
Average	8.45	8.37	.....	8.38	8.37	7.26	7.22	.....	7.22	7.20
Theory	8.47	.....	.....	.....	.....	7.24	.....	.....	.....	.....

[1] Not included in averages, as Mr. Thompson used more iron than was called for in the instructions.
[2] Not included in averages.

NOTES BY THE ANALYSTS.

A. W. Ogden.—The samples seemed a little coarse for nitrogen determination, so I ground same to pass a 0.04 inch sieve. This extra exposure during grinding may have changed the water content a trifle, still I think not notably.

F. Thompson.—The Ulsch-Gunning method was attended with serious frothing and bumping, which could not be readily overcome except by distilling by a current of steam.

J. P. Street.—The Ulsch-Gunning method, while it gives results concordant with the other methods, is very difficult of manipulation. Sample No. 11 gave more

trou e than the other, probably because of the chlorids present. The large amount of material in the flask makes prevention of bumping almost impossible, and in my mind militates strongly against the method.

F. P. Veitch.—Ulsch-Gunning method. This spring I attempted to do some work with this method, comparing it with the regular Gunning for nitrates. I started about 50 samples, but the heavy precipitate caused such violent bumping that more than half of the flasks was broken, and in nearly all other cases soda was carried over.

T. C. Trescot.—

Detailed results of determination of nitrogen in Association of Official Agricultural Chemists' official samples for 1898 by the Ulsch-Gunning method.

Successive distillations.	Serial No. 17239. Referee's No. 11.			Serial No. 17240. Referee's No. 12.	
	Per cent nitrogen.	Per cent nitrogen.	Per cent nitrogen.	Per cent nitrogen.	Per cent nitrogen.
1	8.47	8.37	8.48	7.34	7.23
2	.11	.11	.08	.08	.11
3	.06	.08	.08	.06	.11
4	.06	.08	.00	.00	.00
5	.00	.06	.06	.....	.....
6	.....	.00	.00	.....	.....
Total	8.70	8.70	8.76	7.48	7.45
Average		8.72		7.46	

In the five series of determinations of nitrogen by the Ulsch-Gunning method which are reported above, after the distillation had been completed the residue in the distilling flask was diluted with water and a fresh portion of zinc was added (the quantity usually used for each distillation). The distillation was then repeated, and the quantities of nitrogen indicated under "Distillation No. 2" in the table were obtained. The dilution, addition of zinc, and redistillation were repeated until no more nitrogen came over. In two cases six distillations were necessary before this was accomplished.

It would seem from these results that the reduction of the nitric nitrogen had been incomplete during the treatment with iron, and was completed only by the long treatment with zinc in alkaline solution during the distillation.

The results reported by the modified Kjeldahl in both Nos. 11 and 12 are fairly good, the greatest variation from theory being 0.2 per cent, which occurs in No. 11. Eliminating this result, the greatest difference between the lowest and theory is 0.13 per cent. This is more than it should be, considering that this is one of our standard methods. The results in No. 12 are much better, the widest variation from theory being 0.05 per cent.

With the exception of one set of results, which is about 0.25 per cent higher than theory, what is said about the Kjeldahl method will apply to the Gunning, except that it is slightly lower in the general average. In case of No. 12, eliminating one set of results, which varies from theory about the same as a similar set in No. 11, that is, by 0.25 per cent, the other results by the Gunning are very good.

The results by the Ulsch-Kjeldahl and Ulsch-Gunning methods, with the exception of one set, are very satisfactory. This set exhibits the same difference here that it did in case of the regular official methods. Eliminating this set and comparing the Ulsch-Kjeldahl and Ulsch-Gunning in No. 11, it seems that the results are not quite so good as those obtained by our official methods. With the exception of three results which are about the same as theory, all the others are slightly lower, the lowest being 0.22 per cent below theory; and the general average of all the results is 0.09 per cent below theory in the Ulsch-Kjeldahl and 0.10 in the Ulsch-Gunning method.

The low results given in sample No. 11 by these methods show very clearly that in a sample like this, containing 6.85 per cent of nitric nitrogen, 3.5 grams of iron are not sufficient, and that 4 grams should have been used in order to obtain satisfactory and concordant results. The results obtained by these methods in No. 12 are excellent. The greatest variation between the lowest result and theory is 0.11 per cent, just one-half what it was in the case of No. 11, and the general average is only 0.04 from theory. Of the thirty-two results in sample No. 12 by these methods, only 9 vary more than 0.06 per cent from theory.

It appears from the results with No. 12 that 3.5 grams of iron were certainly sufficient to reduce all the nitrate, and, judging from the results obtained in the case of No. 11, that less than the 3.5 grams would have done; but unfortunately we have no data to prove this.

From the results obtained in our work in these two samples, we ought to be able to lay down the following rule as to the quantity of iron to be used: For every .015 gram of nitric nitrogen present not less than 1 gram of iron should be used. But this point ought, perhaps, to be further investigated.

All the objections to the use of these methods were on account of the bumping caused by the heavy precipitate of iron in the flask. This trouble might be remedied by limiting the quantity of iron to be used in all cases to 2 grams and then regulating the quantity of material to be used by this. Thus, if a fertilizer contain 4 per cent or less of nitric nitrogen, use, say, 0.7 gram, and if more than 4 and less than 7 use 0.5 gram, etc.

On account of sickness the referee was unable to investigate these methods as thoroughly as he had intended; therefore he does not feel justified in making any recommendation as to their adoption.

It would, perhaps, be better to give them another trial.

The work of the determination of the availability of organic nitrogen is a continuation of that of last year, and consists of a further study of the permanganate of potash method in comparison with the pepsin-hydrochloric acid method and a modification of the pepsin method suggested by Mr. Patterson, of Maryland.

In order to carry out this work, ten samples of organic nitrogenous materials were prepared, which were as follows:

No. 1. Dried blood; a low-grade sample.

No. 2. Cottonseed meal.

No. 3. Tankage.

No. 4. Dried fish.

No. 5. Meat scraps.

No. 6. Castor pomace.

No. 7. Steamed leather. This is a sample of the leather used by Dr. Wheeler, of the Rhode Island Station, in his vegetation test.

No. 8. Raw bone.

No. 9. Hoof meal.

No. 10. Dried blood; an average sample. This was also obtained from Dr. Wheeler, and was one of the materials used in his investigation.

As the value of results obtained by chemical methods is dependent upon the relation that these results bear to those obtained by vegetation tests, the referee was anxious to get some of the materials that had been used by the referee of the last year in some vegetation experiments, the results of which were to be ready for publication by this meeting, but, unfortunately, he did not have enough in hand to supply samples.

The materials for samples No. 7 and No. 10 were furnished by Dr. Wheeler. These represent the materials used by him in his vegetation tests upon an acid soil, limed and unlimed. The other samples fairly represent the usual sources of organic nitrogen, and four of these are similar to those used last year.

When the samples and instructions were sent out, the referee requested that the following series of determinations should be made:

(A) *Total nitrogen by Kjeldahl method.*

(B) *Digestion of organic nitrogen by the pepsin-hydrochloric acid method.*

(C) *Digestion of organic nitrogen by the permanganate of potash method.*

(D) *Digestion of organic nitrogen by the modified pepsin-hydrochloric acid method.*

(A) Total Nitrogen by Kjeldahl Method.

Table I.—*Results on total nitrogen by the different analysts.*

Analyst.	Dried blood.	Cottonseed meal.	Tankage.	Dried fish.	Meat scraps.	Castor pomace.	Steamed leather.	Bone meal.	Hoof meal.	Dried blood.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
A. J. Patten, Me. Station	9.91	7.63	6.05	8.27	4.61	5.51	7.07	3.88	15.05	12.97
O. W. Knight, Me. Station	9.93	7.67	6.03	8.38	4.66	5.48	6.96	3.84	14.83	12.95
F. Thompson, N. Y. Station	9.94	7.74	5.92	8.26	[1]4.84	5.45	7.02	3.79	14.72	12.91
	9.84	7.70	5.93	8.29	4.71	5.48	6.99	3.76	14.72	12.81
	9.82	7.66				5.52	6.99	3.82	14.84	12.86
C. H. Jones, Vt. Station	9.90	7.74	5.71	8.36	4.50	5.33	7.00	3.82	14.93	13.08
	9.82	7.64	5.73	8.43	4.48	5.24	7.02	3.75	14.91	12.80
T. C. Trescot, U. S. Dept. of Agr.	9.98	7.44	5.76	8.06	4.60	5.39	6.91	3.82	14.69	12.78
B. L. Hartwell, R. I. Station	9.71	7.53	5.89	8.23	4.62	5.56	6.93	3.88	.....	12.94
Walker Bowman, Louisville Spirit-Cured Tobacco Co	9.97	7.60	5.98	8.52	4.26	5.33	7.14	3.92	15.08	12.80
H. K. Miller, N. C. Station	9.91	[1]7.89	6.05	8.48	4.69	[1]5.67	7.07	3.92	15.01	13.05
	10.02	[1]7.83	6.07	8.51	4.69	5.53	7.08	3.84	14.94	13.05
F. S. Shiver, S. C. Station	10.00	7.68	6.03	[1]8.69	4.76	5.47	7.00	3.89	14.99	12.99
J. P. Street, N. J. Station	9.71	7.51	5.81	8.38	[1]4.37	[1]5.79	6.87	3.85	14.72	[1]12.66
	9.81	7.44	5.78	8.30	[1]4.34	[1]5.74	6.87	3.83	14.67	[1]12.66
	9.76	7.48	5.80	8.34	[1]4.36	[1]5.77	6.87	3.85	14.67	[1]12.67
									14.69	
W. B. Ellett, Va. Station	9.87	7.58	5.80	8.31	4.59	5.31	6.88	3.79	14.67	12.86
	9.82	7.52	5.79	8.26	4.59		6.89	3.81	14.70	12.97
A. T. Eskridge, Va. Station	9.97	7.68	5.80	8.27	4.56	5.31	6.90	3.80	14.70	13.02
Average	9.87	7.60	5.89	8.35	4.62	5.42	6.96	3.83	14.82	12.92

[1] Omitted from the average.

There was only one comment on this part of the work, and that was by Mr. J. P. Street, who says: "I had considerable difficulty in securing good results in the meat scrap, castor pomace, and hoof meal, in a large measure due, I think, to their mechanical condition."

These remarks probably explain the reason for such wide variations in these samples, as they were not as fine as they should have been, but I do not think this reason can be brought against most of the others, and yet the variation is greater in some of them.

In the two samples of blood, the variation was 0.31 per cent in No. 1 and 0.42 per cent in No. 10; in cottonseed meal, 0.45 per cent; in tankage, 0.36 per cent, and in fish, 0.63 per cent.

The meat scraps show a variation of 0.58 per cent; castor pomace, 0.43 per cent, and hoof meal, 0.38 per cent.

(B) Digestion of Organic Nitrogen by the Pepsin-Hydrochloric Acid Method.

The method used in this work was practically the same as that used by the referee on nitrogen for 1896 and 1897, and is given in the proceedings of the association for 1896, page 23.

Table II.—*Results on digestion of organic nitrogen, by different analysts.*

Analysts.	Dried blood.	Cottonseed meal.	Tankage.	Dried fish.	Meat scraps.	Castor pomace.	Steamed leather.	Bone meal.	Hoof meal.	Dried blood.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
C. H. Jones and B. O. White, Vt. Station	1.19	0.70	1.36	2.81	1.78	1.19	3.32	0.07	9.52	1.02
	1.13	.70	1.33	2.90	1.82	1.22	3.29	.07	9.66	.98
T. C. Trescot, U. S. Dept. of Agr	.91	.72	1.43	2.67	1.84	1.22	[1]1.56	.11	.....	[1].72
B. L. Hartwell, R. I. Station	.....	[1]1.00	[1]1.64	[1]3.38	2.03	.....	3.90	.....	.....	.....
H. K. Miller, N. C. Station	.98	.70	1.47	2.90	1.92	1.30	3.36	.11	9.80	[1].70
	.99	.74	1.48	2.88	1.96	1.34	3.37	.13	9.86	.82
F. S. Shiver, S. C. Station	1.07	.79	[1]1.53	2.77	1.90	1.28	3.24	.06	9.80	.96
W. B. Ellett, Va. Station	1.12	.84	1.29	[1]2.46	1.83	1.51	2.85	[1].56	[1]9.06	1.04
	1.00	.76	1.34	[1]2.35	1.79	1.42	3.00	[1].58	[1]9.12	1.00
Average	1.04	.74	1.39	2.82	1.87	1.31	3.29	.09	9.73	.97

[1] Omitted from average.

As a whole, the results by this method are very good with one or two exceptions. One very discordant result obtained in leather may be explained by the following note by Dr. Wiley as to the manner in which the method was carried out in his laboratory:

Inasmuch as our laboratory is in temporary quarters it was not possible to make the determination of pepsin-indigestible nitrogen in precisely the way specified in your circular of instructions. Our results obtained by this method are therefore lacking in value. It will be interesting, however, to note what difference this variation of method has caused in the results.

The digestion period extended for a much longer time than you directed. The samples were weighed and placed in the water bath at 9 a. m. on Wednesday, and the heating was continued for eight hours on that day, when the flasks were removed from the water bath and placed in an ice box. On Thursday the flasks were returned to the water bath at 9 a. m. and the heating continued for eight hours, when they were returned to the ice box. On Friday the flasks were returned to the water bath and heated for seven hours, when they were again returned to the ice box. On Saturday morning the flasks were placed in the water bath and heated for one hour, when their contents were filtered and the determination completed according to your directions.

The 2 cc portions of 10 per cent hydrochloric acid were added to the flasks at the end of the second and fifth hours as you directed. The portions which you directed to be added at the close of the eighth and eleventh hours were added on Thursday at 9 a. m. and at noon, respectively. You will note that the total period of digestion in the water bath is exactly twenty-four hours, as you directed, but that in addition to this the digestion proceeded for forty-nine hours while the flasks were contained in the ice box.

It is worthy of notice that, with the exception of the result in leather and a slight variation in blood, the results obtained under these conditions are excellent.

For convenience of comparison, the referee has calculated the per cent of availability by this method and the data obtained are given in Table No. III.

TABLE III.—*Availability of organic nitrogen.*

Analyst.	Dried blood.	Cottonseed meal.	Tankage.	Dried fish.	Meat scraps.	Castor pomace.	Steamed leather.	Bone meal.	Hoof meal.	Dried blood.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
O. W. Knight, Me. Station	88. 85	[1]91. 13	[1]69. 65	[1]97. 13	[1]52. 36	77. 19	55. 46	[1]39. 59	[1]26. 43	92. 20
		91. 91	[1]67. 66	[1]97. 61	[1]54. 51	77. 19	55. 74	[1]39. 59	[1]28. 19	90. 96
C. H. Jones and B. O. White, Vt. Station	88. 24	90. 90	76. 40	65. 95	59. 91	77. 13	53. 50	98. 15	35. 72	92. 27
T. C. Trescot, U. S. Dept. of Agr.	90. 88	90. 32	75. 17	66. 87	60. 00	77. 37	[1]77. 42	97. 12	Lost.	94. 37
B. L. Hartwell, R. I. Station	.....	[1]86. 72	[1]72. 16	[1]58. 93	56. 06	.....	[1]43. 72	.....	.....	.....
H. K. Miller, N. C. Station	90. 07	90. 84	75. 58	66. 00	58. 64	76. 43	52. 40	96. 91	34. 38	94. 18
F. S. Shiver, S. C. Station	89. 30	89. 71	74. 63	68. 12	60. 08	76. 60	53. 71	88. 46	34. 62	92. 61
W. B. Ellett, Va. Station	89. 23	89. 35	77. 40	71. 10	60. 31	[1]72. 50	57. 23	[1]85. 00	37. 79	92. 15
Average	89. 43	90. 59	75. 72	67. 61	59. 17	76. 99	54. 67	97. 66	35. 63	92. 68
Vegetation cultures	68. 00	74. 00	61. 00	69. 00	.....	72. 77	13. 80	64. 00	67. 00	90. 30

[1] Omitted from average.

The variations noticed in the last table appear much more pronounced here. For instance, bone meal shows a variation in the last table of 0.53 per cent; this becomes in this table a variation of 13 per cent. But even taking this into consideration I do not think we could say that the results are good, and in some material it would appear that this method in the hands of different analysts will not give concordant results.

I should say, perhaps, in justice to the criticisms made in the last table, where it was stated that the results with few exceptions were good, that there appear in this table two sets of results which do not occur in the former one. In the case of blood the results are all that could be expected. Cottonseed meal and castor pomace are good. Dried fish gives an extreme variation of 39 per cent; bone gives 59 per cent, and leather gives 34 per cent. The hoof meal, meat scraps, and tankage are fair. The extreme variation occurring in hoof meal is about 11 per cent. Eliminating the results that cause such wide variation from the averages and comparing these averages with the results obtained by vegetation tests on materials of a similar character, they show very clearly, what is already known in regard to this method, that in such materials as hoof meal and leather it does not give their true availability.

The results obtained under vegetation cultures, except in the case of steamed leather and dried blood, are those obtained by Dr. Jenkins. In the case of leather and sample No. 10 of dried blood the results are from Dr. Wheeler.

(C) DIGESTION OF ORGANIC NITROGEN BY THE PERMANGANATE OF POTASH METHOD.

The pepsin method is exceedingly valuable as a means of detecting inferior forms of organic nitrogen; but one great objection to it is that it consumes a great deal of time, and also that it can not be relied upon entirely as a means of determining the true value of the various forms of nitrogen to plants.

The need, therefore, of a process that will determine quickly and more satisfactorily the availability of organic nitrogen as plant food has led to the suggestion that various solutions of potassium permanganate, alone or with the addition of acid or alkali, be used.

Considerable work was done on these methods last year by the referee and others, which tended to show them unsatisfactory in not giving concordant results. This seems to be the most serious objection.

If some modification of one of these methods can be devised by which this objection can be eliminated, that method would be a very valuable one, especially in regard to time, and from present indications would agree more closely with the vegetation tests than the pepsin method. In the following two tables are found the results

obtained by the method proposed for trial this year. This method was suggested by the referee of last year, and is as follows:

Method: Place 1 gram of sample in a 150 cc flask, add 100 cc of a 2 per cent solution neutral permanganate of potash solution, and place in water bath for thirty minutes. The water bath is kept at the boiling point. At the end of the thirty minutes dilute and filter through a folded filter, wash several times, and determine nitrogen in residue by the Kjeldahl method.

TABLE IV.—*Percentage of nitrogen in residues after digestion with permanganate of potash.*

Analyst.	Dried blood.	Cottonseed meal.	Tankage.	Dried fish.	Meat scraps.	Castor pomace.	Steamed leather.	Bone meal.	Hoof meal.	Dried blood.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
F. Thompson, N. Y. Station	1.58	1.14	0.72	1.35	1.79	1.30	3.62 .56 .72	0.92	2.53 3.53 4.58	1.39
	1.59	1.15	.72	1.41	1.73	1.29	2.80 3.29 3.44	1.06	4.67 6.35 7.20	1.47
C. H. Jones and B. O. White, Vt. Station	.672	.77	.45	.80	.38	1.148	3.57	.966	.81	.69
T. C. Trescot, U. S. Dept. of Agr	.87	1.66	.42	1.46	1.00	1.18	3.26	.86	2.05	2.50
B. L. Hartwell, R. I. Station	.74	1.65	.47	.85	.32	.96	3.55	.56	.....	.71
H. K. Miller, N. C. Station	1.06	1.59	.64	1.41	.63 .67 .91	1.32 1.29	3.74	1.64	2.88	1.87
	1.01	1.66	.73	1.20			3.64	1.47	2.47	2.10
J. P. Street, N. J. Station	1.28	1.88	.89	.99	.93	1.43	3.83	1.06	2.87	1.54
	1.29	1.71	.85	1.12	1.09	1.36	3.71	1.05	2.33	1.65
	1.28	1.79	.87	1.05	1.01	1.39	3.77	1.05	2.60	1.59
F. S. Shiver, S. C. Station	.31	.47	.23	.57	.04	.92	3.02	1.03	1.97	.59
W. B. Ellett, Va. Station	.70 .65	1.85	.63	.89	.42	1.06	3.59	.95	1.84	.87
	.78	1.40	.71	.78	.54	.92	3.35	.78	1.75	.86
A. T. Eskridge, Va. Station	.73 .67	1.62	.60 .74	.89 .71	.44 .45	1.06 .89	3.53 3.48	.89 .84	1.76 1.62	.90 .80

TABLE V.—*Availability of nitrogen as determined by permanganate of potash.*

Analyst.	Dried blood.	Cottonseed meal.	Tankage.	Dried fish.	Meat scraps.	Castor pomace.	Steamed leather.	Bone meal.	Hoof meal.	Dried blood.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
A. J. Patten, Me. Station	91.83	91.63	89.75	90.20	87.65	77.66	55.01	64.96	80.73	89.90
	90.80	89.40	89.43	89.97	84.42	76.20	53.60	61.86		89.05
O. W. Knight, Me. Station	.....	86.96	92.87	89.87	89.04	79.38	53.88	75.26	89.34	95.75
	92.65	85.66	91.21	85.44	89.04	81.57		76.82		96.83
F. Thompson, N. Y. Station	83.87	85.06	87.84	83.33	63.20	77.01	65.71	73.88	67.41	88.88
C. H. Jones and B. O. White, Vt. Station	93.20	89.98	92.13	90.48	91.54	78.26	49.07	74.41	94.57	94.67
T. C. Trescot, U. S. Dept. of Agr	91.28	77.69	92.71	81.89	76.30	78.10	52.82	77.49	86.04	80.44
B. L. Hartwell, R. I. Station	93.38	78.09	92.02	89.67	93.07	82.73	48.77	85.57	.....	94.51
H. K. Miller, N. C. Station	89.57	79.26	88.61	84.70	88.25	76.79	47.88	60.05	82.11	84.75
J. P. Street, N. J. Station	86.90	76.10	85.00	87.40	76.80	75.80	45.10	72.70	82.30	87.40
F. S. Shiver, S. C. Station	96.90	93.88	96.19	93.45	99.16	83.18	56.86	73.52	86.85	95.46
W. B. Ellett, Va. Station	92.78	78.41	88.45	89.87	89.54	81.36	49.64	77.37	87.75	93.27
A. T. Eskridge, Va. Station	92.98	78.90	88.45	90.33	90.13	81.36	49.13	77.37	88.50	93.47

NOTES BY THE ANALYSTS.

A. J. Patten.—Heated the flasks half an hour in steam bath, shook them three times at intervals of ten minutes, washed with about 300 cc of hot water.

O. W. Knight.—In the permanganate method the residue was washed with 400 cc hot distilled water. The material was shaken three times at intervals of ten minutes during the digestion. Found the sample of dried blood very hard to wash after digestion.

L. L. Van Slyke.—The results in 7 and 9 are very variable. You notice that Mr. Thompson took all possible precaution to secure uniformity in treatment. In his hands the method is worthless for these materials.

H. W. Wiley.—In treating sample No. 7 by the permanganate of potash method, it was found at the end of the period of digestion that the permanganate color had been entirely destroyed. Therefore a second determination was made by this method of the indigestible nitrogen in this sample and also in No. 10; but in this case 200 cc of permanganate of potash solution were used instead of 100 cc. Using this large excess of permanganate of potash, the results were very much lowered, viz, 1.40 per cent for sample No. 7 and 0 85 per cent for sample No. 10. The 100 cc portion of permanganate was not entirely discolored in the case of sample No. 10, but the determination was repeated, using the larger quantity, because of the high percentage of indigestible nitrogen found. These results indicate that a variation in the excess of permanganate of potash used causes a very considerable variation in the results obtained.

B. L. Hartwell.—It was interesting to note that in some of the filtrates from the permanganate treatment the color of the permanganate had entirely disappeared. This was noticed in regard to numbers 2, 4, 7, and 10. The filtrate from the leather was brown, and upon testing it with diphenylamin I found that it gave no reaction for nitrates and nitrites. In this respect it differed from the filtrates in the case of the other materials.

H. K. Miller.—In the case of the No. 7, I noticed that all the permanganate was reduced, and then made a determination, using 150 cc instead of 100 cc, and obtained a lower result for nitrogen in the residue.

J. P. Street.—I found it very difficult to remove all the material from the small 150 cc flask, and I attribute my wide results in one or two cases to that exclusively. I also lost some of my leather determination, the flask being too small to allow for the expansion of the liquid in heating.

By an examination of Table IV, it will be seen that there is a great lack of agreement in the results obtained by the different analysts, and in some materials it seems almost impossible to obtain concordant results in the same laboratory.

The variations were so great in both of these tables that it was impossible to attempt to make up the averages. In one or two cases it might have been possible. In cottonseed meal there are thirteen determinations; of this number seven are within 6 per cent of each other, and six are within 3 per cent, but the variation between these two sets is 17 per cent.

If we should eliminate those results which show the greatest variation and average the others, the results would approximate more nearly to the vegetation tests on similar substances, and come nearer to the truth in regard to hoof meal and leather than the pepsin method.

It is possible that the great variation in the method is due to the fact that it is almost impossible to keep all the material down in the solution while the digestion is going on. Some of the substances have a tendency to rise to the top of the solution as soon as the action begins in the flask. If this be shaken down some particles are left on the sides, and these, of course, are not acted upon to the same extent as the rest of the substance. This was noticed especially in case of leather and castor pomace.

This tendency to rise in the flask caused the loss of several determinations of both of the materials in our laboratory, and Mr. J. P. Street reports that he lost several of his determinations in leather on this account. This caused Mr. Street to try the following modification of this method: Instead of the 150 cc flask he used a 500 cc, and carried out the digestion on a steam bath for thirty minutes. The other details were the same. He makes the following remarks in this modification: "As my results show, whether the flask is above the water bath or immersed in the boiling water seems to make little difference; in fact, the (B) results show a slightly higher availability."

These results are as follows:

Substance.	Insoluble nitrogen in permanganate solution.	Availability of nitrogen in permanganate solution.	Substance.	Insoluble nitrogen in permanganate solution.	Availability of nitrogen in permanganate solution.
	Per cent.	*Per cent.*		*Per cent.*	*Per cent.*
Dried blood	1.03, 1.03, 1.03	89.40	Castor pomace	1.54, 1.51, 1.52	73.60
Cottonseed meal	1.54, 1.43, 1.48	80.20	Steamed leather	3.21, 3.48, 3.34	51.30
Tankage	0.85, 0.88, 0.86	85.20	Bone meal	1.34, 1.30, 1.32	65.60
Dried fish	1.08, 1.31, 1.19	85.70	Hoof meal	2.25, 2.28, 2.26	84.60
Meat scrap	0.85, 0.93, 0.89	79.60	Dried blood	1.34, 1.22, 1.28	90.00

Mr. J. M. Bartlett, of the Maine Station, submitted the results of work done by this method in samples of fertilizers, as follows:

No. 1. A fertilizer in which one-half of the nitrogen is from blood and one-half from leather.

No. 2. A fertilizer in which one-half of the nitrogen is from blood and one-half from hoof and bone meal.

No. 3. A fertilizer containing considerable hair and wool.

Nos. 4, 5, 6, 7, and 8 are five samples of fertilizers analyzed in his laboratory.

Number of sample.	Total nitrogen.	Digestible nitrogen by permanganate method.	Number of sample.	Total nitrogen.	Digestible nitrogen by permanganate method.
	Per cent.	*Per cent.*		*Per cent.*	*Per cent.*
1	3.08	65.48, 67.53	5	1.71	76.61, 81.28
2	3.01	91.36, 89.36	6	3.14	81.84, 80.89
3	3.17	86.43, 90.55	7	4.12	94.17, 94.90
4	2.40	77.92, 77.08	8	4.47	93.74, 91.05

Nos. 1 and 2 were made up in the laboratory with dissolved South Carolina rock and the nitrogen from sources mentioned.

The presence of leather as a source of the nitrogen in No. 1 is very clearly brought out in the comparison of the results given in this table for Nos. 1 and 2.

There is another point in the manipulation of this method which it would be well to call attention to, and that is the filtering of the substance after digestion. Some filter very rapidly and well, others are very troublesome, and there is a tendency of the very fine particles to run through the filter. This was noticed in blood, and to some extent in leather and cottonseed meal. This would, of course, be another source of variation in the results.

The various difficulties that were encountered in working with this method, and also the time consumed in filtering and washing, etc., led the referee to try a modification of the alkaline-permanganate method in the hope that it might be possible to eliminate errors and also save time in manipulation. The plan was to

use the same amounts of organic nitrogen with each determination instead of the same amount of material. The solution used consisted of 100 cc of a 3 per cent permanganate solution and 100 cc of a solution of sodium hydrate (containing 600 cc of a saturated solution of sodium hydrate in 1 liter). After the solutions had been added, the flasks were connected with the distilling apparatus and boiled very gently, without distilling any, for thirty minutes. At the end of this time the temperature was increased and the solution distilled as in regular nitrogen work.

Availability of organic nitrogen in alkaline permanganate solution using equal amounts of nitrogen.

Substance.	On a 7 per cent nitrogen basis and 3 per cent permanganate solution.		On a 5 per cent nitrogen basis and 3 per cent permanganate solution.		Availability by vegetation cultures.
	Soluble nitrogen.	Available nitrogen.	Soluble nitrogen.	Available nitrogen.	
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Dried blood	6.64	67.28	8.06	82.27	68.00
	6.93	70.21	8.12	81.66	
Cottonseed meal	3.30	43.42	5.04	66.30	74.00
	3.39	44.61	5.04	66.30	
Tankage	3.64	61.80	4.58	77.76	61.00
	3.40	57.72	4.52	76.74	
Dried fish	4.35	52.10	6.72	80.48	69.00
	4.05	48.50	6.56	78.56	
Meat scraps	3.08	66.67	3.59	77.71	.....
	3.17	68.61	3.49	75.54	.....
Castor pomace	4.06	74.91	4.66	85.98	72.77
	4.04	74.54	4.36	80.44	
Steamed leather	3.26	46.84	3.50	50.29	13.80
	3.02	43.40	3.60	51.72	
Bone meal	2.88	75.20	3.02	78.85	64.00
	2.32	60.60	3.14	81.98	
Hoof meal	11.71	79.01	11.98	80.84	67.00
	11.74	79.22	12.23	82.52	
Dried blood	9.46	73.22	10.36	80.19	90.30
	9.18	71.05	10.33	80.00	

Another series of determinations was run in the same manner, but a 2.5 per cent solution of permanganate was used and a smaller quantity of nitrogen employed.

In the following table are found the two series of results obtained by the modification.

The results in the series of the 3 per cent permanganate solution used were obtained by Mr. W. B. Ellett, and those when the 2.5 per cent solution was used were obtained by Mr. A. T. Eskridge.

The method gave very little trouble in carrying it out.

In one or two cases there was a little bumping at first, but this did not last long. So far as can be judged from the few determinations made, it appears to be possible to obtain good duplicates. The inability to do this has been the chief objection heretofore raised against the alkaline-permanganate method. Whilst the results do not agree very closely with the vegetation tests they do, with one exception (cottonseed meal), show the relative availability of the different forms of nitrogen.

With the results received from the Vermont Station there was also received from Mr. C. H. Jones a series of results obtained by a modification of the alkaline-permanganate method. The permanganate solution used by Mr. Jones consisted of 16

grams of potassium permanganate and 150 grams of sodium hydrate to the liter; 100 cc of this solution were used, and the digestion was for one hour at a temperature below the boiling point. At the end of that time the distillation was allowed to proceed for one hour. The quantity of material used in each case was on a basis of 4.5 per cent of nitrogen.

The results obtained by Mr. Jones are as follows:

Alkaline permanganate in a 4.5 per cent nitrogen basis.

Substance.	Nitrogen content.	Grams used.	Equivalent to nitrogen.	Nitrogen.	Availability.
	Per cent.		*Per cent.*	*Per cent.*	*Per cent.*
Dried blood	9.90	0.45	4.46	2.55	57.2
				2.58	57.8
Cottonseed meal	7.74	.55	4.49	2.08	46.3
				2.03	45.2
Tankage	5.71	.80	4.57	2.52	55.1
				2.59	56.7
Dried fish	8.36	.55	4.60	2.55	55.4
				2.60	56.6
Meat scraps	4.50	1.00	4.50	2.80	62.2
				2.90	64.4
Castor pomace	5.33	.85	4.53	2.47	54.5
				2.51	55.3
Steamed leather	7.00	.65	4.55	1.67	36.7
				1.74	38.2
Bone meal	3.82	1.20	4.58	3.18	69.4
				3.28	71.5
Hoof meal	14.93	.30	4.48	3.15	70.3
				3.15	70.3
Dried blood	13.08	.35	4.58	2.78	60.7
				2.80	61.1

Mr. Jones makes the following comment: "I send you the results obtained by a modification of the alkaline-permanganate method as applied to your samples. Fifty per cent is our dividing line; above 50 we call good and below 50 questionable. Cottonseed meal runs from 45 to 49, but pepsin-hydrochloric acid test at once gives a high availability, which points to the presence of cottonseed meal. Very few of our fertilizers contain cottonseed meal, and the modification need not be condemned simply because it fails on such material, but, as it is quicker as regards time than any of the others, its application can easily sort out all suspicious samples, and these can be tested by the sin method."

These results obtained by the alkaline-permanganate method are exceedingly good, and tend to show that with a further study of it, especially in regard to the strength of the permanganate solution, it may probably become a reliable method.

(D) Digestion of Organic Nitrogen by the Modified Pepsin-Hydrochloric Acid Method.

The method used in this case was one suggested to the referee by Mr. H. J. Patterson, of Maryland. It had been used by him in some digestion experiments in food stuffs in comparison with the regular pepsin method, and he found it gave more concordant results than the pepsin.

It was recommended for trial in order to see if it could be used in work of this kind, and also whether it had any advantage over our regular pepsin method in obtaining better results or in ease of manipulation.

Method: Place 2 grams of sample in a 150 cc flask; add 100 cc of a 0.33 per cent hydrochloric acid solution; add to the solution 0.1 gram of Parke, Davis & Co.'s pulverized pepsin. The flasks are then kept for twelve hours closely corked in a water bath, having a constant temperature of 40° C., with occasional shaking. At the end of the twelve hours the contents are filtered, washed until free from acid, returned to the flask with 100 cc of water, 0.15 gram pancreatin (Merck's), and 0.3 gram of sodium carbonate added, and the whole heated again for twelve hours at 40° C., with occasional shaking. The residue is filtered, thoroughly washed, and the nitrogen determined by the Kjeldahl method.

The results obtained by this method are given in Table VI.

TABLE VI.—*Organic nitrogen insoluble in modified pepsin solution.*

Analyst.	Dried blood.	Cottonseed meal.	Tankage.	Dried fish.	Meat scraps.	Castor pomace.	Steamed leather.	Bone meal.	Hoof meal.	Dried blood.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
C. H. Jones and B. O. White, Vt. Station	0.81	0.18	1.43	2.73	1.65	1.11	0.46	2.28	6.85	0.53
	.67	.25	1.30	2.76	1.62	1.13	.49	2.24	7.70	.51
F. S. Shiver, S. C. Station	.48	.16	1.24	2.18	1.32	1.09	.31	1.74	7.97	.54
A. T. Eskridge, Va. Station	.88	.27	1.00	2.65	1.51	.94	.44	1.93	8.48	.59
	.63	.28	.95	2.74	1.65	.85	.60	1.92	8.49	.66

Availability of organic nitrogen in modified pepsin solution.

Analyst.	Dried blood.	Cottonseed meal.	Tankage.	Dried fish.	Meat scraps.	Castor pomace.	Steamed leather.	Bone meal.	Hoof meal.	Dried blood.
C. H. Jones and B. O. White, Vt. Station	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
	92.49	97.14	76.05	67.26	64.59	79.01	93.15	40.37	51.21	95.98
F. S. Shiver, S. C. Station	95.20	97.92	79.44	74.91	72.27	80.07	95.57	55.27	46.83	96.15
A. T. Eskridge, Va. Station	92.38	96.35	83.10	67.35	65.35	83.05	92.46	49.21	42.24	95.16

Results by this method were received from only three analysts, so that perhaps we are not justified in criticising it.

The results obtained agree fairly well and compare favorably with the regular pepsin method, except in case of leather. With this substance the method is worthless, especially on account of the action of sodium carbonate on leather. In addition to this serious objection, the method is not likely to be used very often on account of the trouble of manipulation, requiring, as it does, two digestion periods of twelve hours each, making it very inconvenient to attend to, and also two filterings. The filtering is very slow, in some cases requiring two or three filters and one or two days' time.

This subject of the availability of organic nitrogen has been very thoroughly studied at the Vermont Station, a large amount of work having been done with the permanganate of potash method, especially the alkaline permanganate.

The materials used represent samples from nearly all the sources of organic nitrogen, as well as samples of fertilizers containing various mixtures. The results of these investigations are published in the last annual report of the station. A copy of this part of the report is at hand and can be seen by anyone wishing to do so.

The only recommendation which your referee wishes to make on the subject is that the permanganate of potash methods for the determination of the availability of organic nitrogen be further studied next year, especially some modification of the alkaline-permanganate method.

After twenty-four hours digestion, filter, wash four or five times, using 20 to 30 cc of water for each washing, and determine nitrogen by the Kjeldahl method in the washed and dried residue.

PERMANGANATE OF POTASH METHOD.

Weigh 1 gram of sample into a 150 cc flask, add 100 cc of a 2 per cent neutral permanganate of potash solution, and place in a water bath for thirty minutes. The water bath is kept at the boiling point. At the end of the thirty minutes dilute and filter through a folded filter, wash several times, and determine nitrogen in residue by Kjeldahl method.

MODIFIED PEPSIN-HYDROCHLORIC ACID METHOD.

Weigh 2 grams of sample into a 150 cc flask, add 100 cc of a 0.33 per cent hydrochloric acid solution; add to this 0.1 gram of scale pepsin. The flasks are then kept for twelve hours loosely corked in a water bath having a constant temperature of 40° C., with occasional shaking. At the end of the twelve hours the contents are filtered, washed until free from acid, returned to the flask with 100 cc of water, 0.15 gram pancreatin (Merck's), and 0.3 gram of sodium carbonate added, and the whole heated again for twelve hours at 40° C., with occasional shaking. The residue is filtered, thoroughly washed, and the nitrogen determined by the Kjeldahl method.

At the conclusion of the reading of the report the president called for any paper on the subject. None being offered, the discussion of the subject was declared to be in order.

Mr. C. H. Jones reported results of his experiments in leather, dried blood, etc. His experience led him to believe that the nitrogen in leather in commercial fertilizers could not be detected in quantities of less than 2 per cent. When leather is treated with sulphuric acid, which is the usual custom before mixing it in fertilizers, its texture is practically destroyed so as to render its detection extremely difficult. He thought the permanganate method valuable in showing those samples which fall below 50 per cent of available nitrogen, and these could be set aside for further testing with the pepsin method.

Mr. H. J. Wheeler stated that in comparative tests of the availability of leather and dried blood in vegetation experiments he had found that about 19 per cent of the leather and 90 per cent of the blood were available for plant nutrition.

Mr. Jones thought it was hardly reasonable to require a method which would give the same results as vegetation experiments.

Mr. J. A. Myers asked Mr. Wheeler if there were any means of determining the degree of pulverization of the leather.

Mr. Wheeler replied that it was difficult to determine the degree of pulverization of the leather. There should not be so great a discrepancy between the methods. This sample was further pulverized before it was sent out, but in the vegetation tests it was not pulverized.

The PRESIDENT. The recommendations of the referee on nitrogen will be referred to the committee.

The president then called for the report of the referee on potash. The report was read by the referee, Mr. C. H. Jones.

REPORT ON POTASH.

By C. H. Jones, *Referee*, and B. B. Ross, *Associate Referee*.

The methods for estimating potash have been quite thoroughly studied by this association in years past, so far as potash salts and fertilizers are concerned. The applicability of these methods to wood ashes and cotton-hull ashes being deemed of considerable importance, the referee for 1898 was directed to investigate our present methods for determining K_2O as applied to these materials.

PREPARATION OF SAMPLES.

Two samples were accordingly prepared; No. 1, consisting of high grade wood ashes obtained from Waterbury Center, Vt., and No. 2, cotton-hull ashes from a Connecticut dealer. Particular care was used in collecting and storing the ashes from which No. 1 was taken, and results show this must have been the case. A lower grade sample containing more silicate of potash would have been preferable for our work.

The finely ground samples were carefully and independently mixed by two men, and then bottled and sealed. Three samples from each lot, selected at random after bottling, were analyzed in the Vermont laboratory. The uniform results obtained would indicate perfect mixing of the original materials.

DIRECTIONS FOR WORK.

Under date of January 24, 1898, circular letters were sent to interested parties, stating scope of work and requesting cooperation. Favorable replies were received from twenty chemists and samples were sent them with instructions as follows:

Agricultural Experiment Station, *Burlington, Vt.*

Dear Sir: At the last meeting of the Association of Official Agricultural Chemists, the referee on potash was directed to test our present methods for determining potash as applied to wood ashes and cotton-hull ashes.

Under separate cover we send you by mail samples Nos. 1 and 2. You are requested to determine the potash in them, following instructions here given.

Determine moisture on 2 grams of the material as per page 11, Bulletin 46, United States Department of Agriculture, Division of Chemistry.

1. *Water soluble potash.*—(*a*) Determine according to the Lindo-Gladding method for fertilizers described on page 2, Circular No. 4, United States Department of Agriculture, Division of Chemistry.

(*b*) Determine as per optional method on pages 2 and 3 of Circular No. 4.

2. *Acid soluble potash.*—(*a*) Method of making solution: Place 5 grams of the material in a 250 cc flask, moisten with about 20 cc H_2O, and cautiously add 20 cc of HCl, sp. gr. 1.20. When the CO_2 is nearly expelled add about 50 cc of H_2O and boil for 30 minutes.

(*b*) Partially cool, add slight excess of ammonia and sufficient powdered ammonium oxalate to precipitate all the lime present. Cool and make to 250 cc. Filter through a dry filter and determine K_2O as per Lindo-Gladding method, page 2, Circular No. 4.

(*c*) Make solution as just directed by treating 5 grams of the material with HCl and boiling. Do not add ammonia and ammonium oxalate. Dilute with H_2O, cool, and make to volume of 250 cc. Filter through a dry filter and determine K_2O as under the optional method, page 3, Circular No. 4, using 25 cc of the solution.

3. Make blank determinations on reagents, using amounts as required in the actual analysis.

4. Owing to the danger of the final K_2PtCl_6 being contaminated with silicic acid you are requested to make sure that the K_2PtCl_6 obtained is soluble in water, by dissolving the salt, drying and reweighing the crucible. Use Gooch crucibles for filtering the K_2PtCl_6.

5. In reporting results kindly state (1) amount of material employed, (2) total weight of K_2PtCl_6 found, (3) weight of K_2PtCl_6 from reagents, (4) increase due to silicic acid, if any, (5) per cent of moisture found.

Report results not later than September 1, 1898.

Yours, truly,

C. H. Jones, *Referee.*
B. B. Ross, *Associate.*

RESULTS OBTAINED.

Results have been received from the following chemists: Mr. A. W. Ogden, Experiment Station, New Haven, Conn.; Mr. C. C. McDonnell, Mr. John Thompson, and Mr. F. S. Shiver, Clemson College, South Carolina; Mr. F. P. Veitch, College Park, Md.; Mr. G. E. Patrick, United States Department of Agriculture, Washington, D. C.; Mr. Walker Bowman, chemist for the Louisville Spirit-Cured Tobacco Company, Louisville, Ky.; Mr. B. O. White and Mr. C. H. Jones, Vermont Experiment Station, Burlington, Vt.

The following table shows in detail the results obtained. The percentage of K_2O was calculated in all cases by the referee using the factor .1939. In no case was correction made for bulk of undissolved and precipitated material.

Results of potash work for 1898.

WATER SOLUBLE POTASH, WOOD ASHES, No. 1.

Analyst.	Moisture.	Quantity used.	K_2PtCl_6 and impurities.		K_2PtCl_6 in reagents.		Increase due to silica, etc.		Corrected weight, K_2PtCl_6		Potassium oxid.	
			Lindo-Gladding.	Optional.	Lindo-Gladding.	Optional.	Lindo-Gladding.	Optional.	Lindo-Gladding.	Optional.	Lindo-Gladding.	Optional.
	P.ct.	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*P.ct.*	*P.ct.*
A. W. Ogden, New Haven, Conn	0.18	1	0.4855	0.4879	0.0000	0.0026	0.0006	0.0010	0.4849	0.4843	9.40	9.39
	.18	1	.4851	.4888	.0000	.0026	.0005	.0020	.4846	.4842	9.40	9.39
C. C. McDonnell, Clemson College, S. C	.29	1	.4836	[1].2601	.0003	.0198	[2]	.....	.4833	[1].2403	9.37	9.32
John Thompson, Clemson College, S. C	.26	½	.2406	.2475	.0003	.0056	.0014	.0009	.2389	.2410	9.26	9 34
F. S. Shiver, Clemson College, S. C	.21	½	.2470	.2692	.0029	.0254	.0045	.0039	.2396	.2399	9.29	9.30
F. P. Veitch, College Park, Md	.07	1	.4863	.....	.0020	.....	.....	.....	.4843	.....	9.39	.....
	.08	1	.4889	.....	.0020	.....	.....	.....	.4869	.....	9.44	.....
B. O. White, Burlington, Vt.	.30	1	.4858	.....	.0000	.....	.0010	.....	.4848	.....	9.40	.....
	.26	1	.4842	.....	.0000	.....	.0005	.....	.4837	.....	9.38	.....
C. H. Jones, Burlington, Vt.	.27	1	.4850	.4893	.0000	.0056	.0000	.0016	.4850	.4821	9.40	9.35
	.25	1	.4860	.4922	.0000	.0056	.0005	.0012	.4855	.4854	9.41	9.41
G. E. Patrick, Washington, D. C	[3].02	1	.4889	.5080	.0011	.0080	.....	.....	.4878	.5000	9.46	[4]9.69
	[3].10	1	.4875	.....	.0011	.....	.....	.....	.4864	.....	9.43	.....
Walker Bowman, Louisville, Ky	.22	1	[5].4536	[5].4895	.0000	.0277	.....	.....	[5].4536	[5].4613	[4]8.79	[4]8.95
Average	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	9.39	9.36

WATER SOLUBLE POTASH, COTTON-HULL ASHES, No. 2.

A. W. Ogden, New Haven, Conn	4.51	½	0.4344	0.4412	0.0000	0.0026	0.0000	0.0035	0.4344	0.4351	16.84	16.87
	4.46	½	.4361	.4385	.0000	.0026	.0000	.0020	.4361	.4339	16.91	16.82
C. C. McDonnell, Clemson College, S. C	4.17	½	.4291	.4508	.0003	.0198	[2]	.....	.4288	.4310	16.63	16.72
John Thompson, Clemson College, S. C	4.30	½	.4308	.4357	.0003	.0056	.0010	.0012	.4295	.4289	16.65	16.64

[1] One-half gram material used.
[2] Filtered before adding K_2PtCl_6.
[3] After 5¼ hours' drying.
[4] Omitted from average.
[5] Results calculated to a 1 gram basis by referee.

Results of potash work for 1898—Continued.

WATER SOLUBLE POTASH, COTTON-HULL ASHES, No. 2—Continued.

Analyst.	Moisture.	Quantity used.	K_2PtCl_6 and impurities.		K_2PtCl_6 in reagents.		Increase due to silica, etc.		Corrected weight, K_2PtCl_6		Potassium oxid.	
			Lindo-Gladding.	Optional.	Lindo-Gladding.	Optional.	Lindo-Gladding.	Optional.	Lindo-Gladding.	Optional.	Lindo-Gladding.	Optional.
	P. ct.	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*P. ct.*	*P. ct.*
F. S. Shiver, Clemson College, S. C	4.72	½	.4388	.4582	.0020	.0169	.0000	.0035	.4368	.4378	16.94	16.98
F. P. Veitch, College Park, Md	[1]3.92	½	.4675	.....	.0020	.....	.....	.....	.4655	.....	[2]18.05	.....
	[1]4.00	½	.4650	.....	.0020	.....	.....	.....	.4630	.....	[2]17.95	.....
B. O. White, Burlington, Vt.	4.28	½	.4364	.....	.0000	.....	.0000	.....	.4364	.....	16.92	.....
	4.34	½	.4330	.....	.0000	.....	.0000	.....	.4330	.....	16.79	.....
C. H. Jones, Burlington, Vt.	4.45	½	.4360	.4398	.0000	.0056	.0000	.0014	.4360	.4328	16.91	16.78
	4.42	½	.4350	.4428	.0000	.0056	.0005	.0020	.4345	.4352	16.85	16.88
G. E. Patrick, Washington, D. C	[3]4.25	1	.9187	[4]	.0011	.....	.....	.....	.9176	[4]	[2]17.79	.....
	[3]4.20	1	.9220	.....	.0011	.....	.....	.....	.9209	.....	[2]17.85	.....
Walker Bowman, Louisville, Ky	0.88	1	[5].8228	[5].8531	.0000	.0482	.....	.....	[5].8228	[5].8049	[2]15.95	[2]15.60
Average	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	16.83	16.81

ACID SOLUBLE POTASH, WOOD ASHES, No. 1.

A. W. Ogden, New Haven, Conn	0.18	1	0.5113	0.5164	0.0000	0.0026	0.0000	0.0017	0.5113	0.5121	9.91	9.93
	.18	1	.5135	.5156	.0000	.0026	.0000	.0017	.5135	.5113	9.96	9.91
C. C. McDonnell, Clemson College, S. C	.29	½	.2568	.2733	.0003	.0151	[6]	.....	.2565	.2582	9.95	10.01
John Thompson, Clemson College, S. C	.26	½	.2605	.2680	.0003	.0065	.0014	.0010	.2588	.2605	10.03	10.10
F. S. Shiver, Clemson College, S. C	.21	½	.2675	.2916	.0044	.0325	.0056	.0053	.2575	.2538	9.98	9.84
F. P. Veitch, College Park, Md	.07	1	.5012	.....	.0020	.....	.0000	.....	.4992	.....	9.68	.....
	.08	1	.4974	.....	.0020	.....	.0000	.....	.4954	.....	9.61	.....
B. O. White, Burlington, Vt.	.30	½	.2572	.....	.0000	....	.0000	.....	.2572	.....	9.97	.....
	.26	1	.5125	.....	.0000	.....	.0000	.....	.5125	.....	9.94	.....
C. H. Jones, Burlington, Vt.	.27	½	.2555	.....	.0000	.....	.0000	.....	.2555	.....	9.91	.....
	.25	1	.5152	.5237	.0000	.0091	.0000	.0015	.5152	.5131	9.99	9.95
	.....	1	.....	.5229	.....	.0091	.....	.0022	.....	.5116	.....	9.92
G. E. Patrick, Washington, D. C	[3].02	1	.4935	.5116	.0011	.0118	.....	.....	.4924	.4998	[2]9.54	9.69
	[3].10	1	.4916	.....	.0011	.....	.....	.....	.4905	.....	[2]9.51	.....
Walker Bowman, Louisville, Ky	.22	1	[5].4688	[5].5110	.0000	.0283	.....	.....	[5].4688	[5].4827	[2]9.09	[2]9.36
Average	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	9.90	9.92

[1] At end of ten five-hour periods of drying found 5.12 and 5.17 per cent moisture.
[2] Omitted from average.
[3] After 5½ hours' drying.
[4] Results unsatisfactory and no time to repeat.
[5] Results calculated to a 1 gram basis by referee.
[6] Filtered before adding K_2PtCl_6.

Results of potash work for 1898—Continued.

ACID SOLUBLE POTASH, COTTON-HULL ASHES, No. 2.

Analyst.	Moisture.	Quantity used.	K_2PtCl_6 and impurities.		K_2PtCl_6 in reagents.		Increase due to silica, etc.		Corrected weight, K_2PtCl_6		Potassium oxid.	
			Lindo-Gladding.	Optional.	Lindo-Gladding.	Optional.	Lindo-Gladding.	Optional.	Lindo-Gladding.	Optional.	Lindo-Gladding.	Optional.
	P. ct.	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*Gr.*	*P. ct.*	*P. ct.*
A. W. Ogden, New Haven, Conn	4.51	½	0.5420	0.5423	0.0000	0.0026	0.0010	0.0009	0.5410	0.5388	20.98	20.90
	4.46	½	.5431	.5434	.0000	.0026	.0006	.0002	.5425	.5406	21.04	20.96
C. C. McDonnell, Clemson College, S. C	4.17	½	.5434	.5615	.0003	.0151	[1]	.....	.5431	.5464	21.07	21.19
John Thompson, Clemson College, S. C	4.30	½	.5475	.5549	.0003	.0065	.0012	.0012	.5460	.5472	21.17	21.22
F. S. Shiver, Clemson College, S. C	4.72	½	.5456	.5804	.0038	.0325	.0012	.0049	.5406	.5430	20.96	21.06
F. P. Veitch, College Park, Md	[2]3.92	½	.5365	.....	.0020	.....	.0129	.....	.5216	.....	[3]20.23	.....
	[2]4.00	½	.5370	.....	.0020	.....	.0129	.....	.5221	.....	[3]20.25	.....
	.....	½	.5318	.....	.0020	.....	.0129	.....	.5169	.....	[3]20.05	.....
B. O. White, Burlington, Vt.	4.28	½	.5395	.....	.0000	.....	.0006	.....	.5389	.....	20.90	.....
	4.34	½	.5436	.....	.0000	.....	.0012	.....	.5424	.....	21.03	.....
C. H. Jones, Burlington, Vt	4.45	½	.5425	.5517	.0000	.0091	.0000	.0012	.5425	.5404	21.04	20.95
	4.42	½	.5448	.5490	.0000	.0091	.0010	.0008	.5438	.5391	21.08	20.91
G. E. Patrick, Washington, D. C	[4]4.25	1	1.0927	1.0952	.0011	.0118	.....	.....	1.0916	1.0834	21.16	21.01
	[4]4.20	1	1.0946	.....	.0011	.....	.....	.....	1.0935	.....	21.20	.....
Walker Bowman, Louisville, Ky	.88	½	[5].5100	[5].5190	.0000	.0233	.....	.....	[5].5100	[5].4957	[3]19.78	[3]19.22
Average	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	21.06	21.03

[1] Filtered before adding K_2PtCl_6.
[2] At end of ten five-hour periods of drying found 5.12 and 5.17 per cent moisture.
[3] Omitted from average.
[4] After 5¼ hours' drying.
[5] Calculated to one-half gram basis by referee.

COMMENTS BY THE REFEREE.

A comparison of the average results by both methods shows practically no difference, the extreme variation being only 0.03 per cent. Forty-four results are used in making the averages for the Lindo-Gladding method, and thirty for the optional.

Under water soluble potash No. 1, thirteen out of fourteen results by the Lindo-Gladding method vary but 0.20 per cent (9.26–9.46 per cent, with an average of 9.39 per cent). Seven out of nine results by the optional method agree closely with each other, the variations being but 0.11 per cent (9.30–9.41 per cent, average 9.36 per cent).

In sample No. 2 the variations by each method are larger than in the preceding case. Nine out of fourteen give an average of 16.83 per cent by the Lindo-Gladding method; highest 16.94, lowest 16.63 per cent, difference 0.31 per cent K_2O; while by the optional seven out of eight show a variation of 0.34 per cent, highest 16.98, lowest 16.64 per cent, average 16.81 per cent K_2O.

The acid soluble potash results show far more uniformity than was expected. With No. 1, eleven out of fourteen agree within 0.42 per cent K_2O (9.61–10.03 per cent, average 9.90 per cent K_2O) by the Lindo-Gladding method, and within 0.41 by the optional (9.69–10.10 per cent, average 9.92 per cent K_2O).

No. 2, Lindo-Gladding method, shows eleven out of fifteen to vary 0.30 per cent (20.90-21.20 per cent, average 21.06 per cent K_2O). Optional method, eight out of nine vary 0.32 per cent; highest 21.22 per cent, lowest 20.90 per cent, average 21.03 per cent K_2O.

Table summarizing the foregoing data.

	Lindo Gladding.					Optional.					Average per cent K_2O.	
	Number of results received.	Number averaged.	Highest per cent K_2O.	Lowest per cent K_2O.	Extreme variation, per cent K_2O.	Number of results received.	Number averaged.	Highest per cent K_2O.	Lowest per cent K_2O.	Extreme variation, per cent K_2O.	Lindo-Gladding.	Optional.
Wood ashes, water soluble potash	14	13	9.46	9.26	0.20	9	7	9.41	9.30	0.11	9.39	9.36
Cotton-hull ashes, water soluble potash	14	9	16.94	16.63	.31	8	7	16.98	16.64	.34	16.83	16.81
Wood ashes, acid soluble potash	14	11	10.03	9.61	.42	9	8	10.10	9.69	.41	9.90	9.92
Cotton-hull ashes, acid soluble potash	15	11	21.20	20.90	.30	9	8	21.22	20.90	.32	21.06	21.03

The chemist reporting low results throughout apparently did not follow the prescribed methods. It is also to be regretted that the high results by the Lindo-Gladding method on sample No. 2 were not checked by the optional method. In one case it was not attempted, and in the other unsatisfactory results were reported with no time to repeat.

Your attention is now directed to the columns showing corrections made, both because of potash in reagents used and of increase due to silica, etc. The former, in the case of the Lindo-Gladding method, sample No. 1, shows an extreme of 8.8 mg K_2PtCl_6, equivalent to 0.17 per cent K_2O, and in No. 2 of 7.6 mg K_2PtC_6, or 0.15 per cent K_2O. The latter furnishes an insoluble residue correction varying from nothing to 11.2 mg K_2PtCl_6, or 0.22 per cent K_2O, if we except the results of one chemist whose large correction seems to be due to an insufficient washing in the final dissolving of the potassium salt.

When the optional method was used the K_2PtCl_6 found in reagents varied from 2.6 to 65 mg, or from .05 to 1.26 per cent K_2O. The same tendency toward large corrections has been observed in past years when this method has been employed. The increase due to silica, etc., by the optional method does not differ materially from that obtained by the Lindo-Gladding.

DIRECT EVAPORATION WITH K_2PtCl_6.

It was suggested to your referee last year by Mr. Winton that good results might be obtained in water soluble potash determinations on materials similar to those under consideration, by directly evaporating the water solution, without the addition of NH_4OH and $(NH_4)_2C_2O_4$, with K_2PtCl_6, the final precipitate being washed with NH_4Cl solution as usual. A similar method of procedure was adopted for the analysis of kainit at our 1897 meeting.

Mr. Ogden, of the Connecticut Station, kindly sent results by this method, which, together with those obtained by Mr. White and myself, are found in the following table:

Results in determination of potash.

Analyst.	No. 1.			No. 2.		
	Grams used.	K_2PtCl_6 found.	K_2O found.	Grams used.	K_2PtCl_6 found.	K_2O found.
		Grams.	*Per cent.*		*Grams.*	*Per cent.*
A. W. Ogden, Connecticut	1	0.4878	9.46	½	0.4342	16.84
					.4353	16.88
B. O. White, Vermont	1	.4860	9.42	½	.4362	16.91
C. H. Jones, Vermont	1	.4855	9.41	½	.4347	16.86

MOISTURE DETERMINATIONS.

Some interesting data were furnished under this head by Mr. G. E. Patrick, who found the loss of moisture after five hours' drying to be 0.02 and 0.10 per cent with sample No. 1. Continued drying showed a gradual increase in weight, due doubtless to absorption of CO_2, until after forty-one hours' drying, gains in weight of 1.11 and 1.07 per cent were recorded.

The same chemist, with sample No. 2, observed a gradual loss in weight, and the moisture percentages at the end of forty-one hours' drying were 7.28 and 7.17.

A similar behavior with No. 1 was observed in the Vermont laboratory, but the drying was not carried so far. Our reported results represent the loss after five hours' drying at 100° C. and continued heating for one hour did not change them.

CHLORIN IN FERTILIZERS.

A second duty of the referee was to investigate methods for determining chlorin. This, as I understand it, refers to a quick and reliable means of ascertaining whether or not the potash present in fertilizers is in the form of chlorid, or agriculturally what amounts to the same thing, whether sufficient chlorin exists in the material to account for all the potash as such.

From conversation with members of this association in 1897, I learned that among the methods in use was the volumetric estimation of the chlorin by standard $AgNO_3$ in an aliquot of the soluble phosphoric acid solution, or in solutions specially prepared, and the regular gravimetric method.

I desire to present for your consideration a method which was introduced in our laboratory by Mr. Hills in the spring of 1896. With a slight modification it has been used, successfully, we think, for the past three seasons on all our commercial fertilizer samples. It consists in adding to 50 cc of the Lindo-Gladding potash solution (equivalent to 1 gram material) an excess of HNO_3 and as many cc of an $AgNO_3$ solution (1 cc of which corresponds to the chlorin equivalent to 1 per cent of K_2O on a basis of 1 gram material) as the percentage of K_2O found exceeds 0.5 per cent. The solution is boiled for about five minutes and stirred as in the regular chlorin determination. The precipitated AgCl is allowed to settle and a small portion of the supernatant liquid is decanted into a test tube. This is roughly divided into two portions; to one is added a tiny fragment of crystalline AgNO , to the other a few drops of dilute HCl or NH_4Cl solution. A precipitate in the first case indicates an excess of chlorin, even for 0.5 per cent more K_2O than the amount already found to be present, while a precipitate in the second shows an excess of $AgNO_3$, and consequently an insufficiency of chlorin. In the latter case, which with us amounted to less than 20 per cent of the entire number of fertilizers collected in 1898, the AgCl is filtered through a tared Gooch crucible, washed, dried, and weighed as usual. The weight of $AgCl \times 100 \div 3$ gives the per cent of K_2O present as chlorid and is sufficiently accurate for general purposes.

The $AgNO_3$ solution is prepared by dissolving 36.17 grams of c. p. crystalline $AgNO_3$ in water and making to bulk of one liter.

To test the accuracy of the method three fertilizer mixtures were prepared. No. 1 contained potash in the form of common high-grade sulphate, No. 2 had its potash in the form of muriate, and No. 3 contained 4 parts of muriate and 6 parts of sulphate.

Analysis showed them to contain: No. 1, 4.24 per cent; No. 2, 4.40 per cent, and No. 3, 4.32 per cent of K_2O. $AgNO_3$ solution added, in cc equivalent to the above percentages of K_2O, to 50 cc of the Lindo-Gladding solution, treated as directed, filtered, dried, and weighed, gave weights of AgCl corresponding to 0.54 per cent, 4.25 per cent, and 2.64 per cent, respectively, of K_2O as chlorid or perhaps chlorin in other combinations sufficient to account for the equivalent of K_2O just stated. Tested qualitatively as per method, No. 2 showed an excess, while Nos. 1 and 3 an insufficiency of chlorin. The total chlorin in No. 2 amounted to an equivalent of 5.74 per cent K_2O.

This method has the merit of great rapidity and, for the purposes stated, is sufficiently accurate, quickly disposing of the vast majority of samples containing an excess of chlorin, the only question in most cases being simply this: Is there or is there not an amount of chlorin present equivalent to the potash percentage found?

We have tested, so far as time and opportunity would permit, the volumetric titration of chlorin in (1) soluble phosphoric acid, (2) Lindo-Gladding potash, and (3) specially prepared water solutions. Results have, as a rule, been satisfactory, but far more time and attention were required than when the method previously stated was used.

RECOMMENDATIONS.

I would recommend that the Lindo-Gladding method be adopted for determining water soluble potash in wood ashes and cotton-hull ashes, special attention being paid to the words, "The precipitate should be perfectly soluble in water," found in the description of this method, page 2, Circular No. 4, United States Department of Agriculture, Division of Chemistry.

I would also suggest that Gooch crucibles be used for filtering the K_2PtCl_6 when the Lindo-Gladding method is employed.

It is the opinion of your referee that the optional method should not be used unless blank determinations on reagents are made along with it.

At the conclusion of the reading of the report of the referee on potash, the president called for papers. None being presented, the general discussion was in order.

Mr. B. B. Ross called attention to his experience with asbestos and Gooch filters, to the effect that asbestos is attacked slightly by ammonium chlorid solution.

Mr. Jones replied that when asbestos had been previously treated with hydrochloric acid no loss from ammonium chlorid solution had been noticed, but some fibers may be carried through mechanically.

Mr. Ross stated that he used an ashless filter.

Mr. Wheeler asked Mr. Ross how he treated his asbestos before using it for filtering purposes.

Mr. Ross described his method of preparing the asbestos.

Mr. J. L. Hills suggested that manufactured asbestos paper might perhaps be used for filtering purposes.

Mr. Ross stated that he had used three or four different samples of this paper and always found that it suffered loss. He hoped other members would try this further.

After some discussion of the subject of asbestos felt and filters, the president stated that there were still ten minutes and perhaps there would be time for some miscellaneous business.

The secretary made announcements in regard to local arrangements for the entertainment of the members, and especially that the Cosmos Club had extended its courtesies to the members of the convention during the time of their stay here. He asked the members to make the Cosmos Club their headquarters, where they would find stationery, newspapers and magazines, and other comforts of life.

The president called upon Dr. Wheeler to report what action had been taken by him and his associates in regard to the criticism of the methods of potash analysis of our association on the part of certain German chemists.

Dr. Wheeler reported that he had offered an article to Dr. Nobbe, editor of the Versuchsstationen Journal, but he had not received any response. He had also called upon Dr. Nobbe during the past summer. It was evident it was not desired to publish the article in the Journal of the Versuchsstationen. He and his associates, therefore, thought it wise to offer it to The Chemical News, where it had been published, and copies of the reprint could be had by members of the association.

The reprints were distributed to such members of the association as desired them.

At the conclusion of Dr. Wheeler's oral report on this subject, the association adjourned for luncheon.

FRIDAY—AFTERNOON SESSION.

On the reassembling of the association, the president called for the report of the referee on phosphoric acid. Mr. B. W. Kilgore presented the following report:

REPORT ON PHOSPHORIC ACID.

By B. W. Kilgore, *Referee.*

The main object of the work on phosphoric acid the past year has been to further test the volumetric method, which this association has had under consideration for several years, in comparison with the official gravimetric one for the estimation of phosphoric acid in various materials. A second object of the work was a test of the Wagner citrate method for the estimation of available phosphoric acid in slag meals. So few results were received on the latter subject that it is not considered worth while to report them. The present report is therefore limited to the first named subject.

Five samples, with the following outline for investigation, were sent to the chemists desiring to take part in the work.

Instructions for phosphoric acid work for the Association of Official Agricultural Chemists for 1898.

Five samples have been prepared for use in the investigation of the methods for determining phosphoric acid for the Association of Official Agricultural Chemists for 1898.

No. 1 is the finer portion of cottonseed meal.

No. 2 is fine-ground South Carolina phosphate.

No. 3 is a chemically pure phosphate, containing about 20 per cent phosphoric anhydrid. The specimen tube contains 10 grams of the salt, accurately weighed, which should be dissolved in 1 liter of water. One hundred cc. of the solution will contain 1 gram of the phosphate.

No. 4 is fine-ground European slag meal.

No. 5 is fine-ground American slag meal.

Please determine total phosphoric acid in all five samples:

I. By the official gravimetric method (Bulletin 46, Chemical Division, United States Department of Agriculture, p. 12).

II. By the volumetric method as described at end of report.

III. Determination of available phosphoric acid by the Wagner citrate method. For this work use samples Nos. 4 and 5.

The following is a portion of the outline made by Drs. Wiley and McDonnell for the work on this subject last year, which you are requested to follow:

The Wagner citrate method.—Five grams of slag meal, without further grinding or sifting, are put into a half-liter flask and filled to the mark with dilute ammonium citrate solution, the temperature of which is 17°. The flask is closed by means of a rubber stopper, and immediately put into a revolving agitating apparatus for thirty minutes, the revolutions being made at the rate of 30 or 40 per minute. Filter at once; 50 cc of the filtrate are mixed with 100 cc of the molybdate solution and heated on the water bath at from 80° to 90° for from ten to fifteen minutes. Remove from bath and let cool to the temperature of the room and proceed in the usual way.

Wagner's concentrated ammonium citrate solution contains in 1 liter 150 grams crystallized citric acid and 23 grams of ammoniacal nitrogen. The latter is to be determined accurately by distilling the ammonia from a measured volume of the solution (after making the solution up with approximately the proper amount of ammonia to eight or nine tenths of the intended volume). The determination is made as in the Gunning method, omitting the digestion. The solution is now made up with water so that the total nitrogen per liter is 23 grams.

For use, two volumes of this solution are diluted with three volumes of water.

For further details of this method see Wiley's Agricultural Analysis, Volume II.

Results on other samples and points in the methods outlined or on subjects relating to phosphoric acid will be gladly received and credit given in the report. A plentiful supply of samples is on hand and more can be had if desired.

Please report results of your work at your convenience but in time to make up report for the association meeting.

B. W. KILGORE, *Referee.*

RESULTS BY GRAVIMETRIC AND VOLUMETRIC METHODS.

Twenty-two chemists took part in the work and the results by the two methods on the samples sent out by the referee are brought together in Table I.

TABLE I.—*Total phosphoric acid by gravimetric and volumetric methods, referee's samples.*

Analyst.	No. 1. Cottonseed meal.		No. 2. South Carolina phosphate.		No. 3. Disodium hydrogen phosphate.		No. 4. European slag.		No. 5. American slag.	
	Gravimetric method.	Volumetric method.	Gravimetric method.	Volumetric method.	Gravimetric method.	Volumetric method.	Gravimetric method.	Volumetric method.	Gravimetric method.	Volumetric method.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
W. L. Mitchell, Conn. Exp. Station.	2.92	2.97	27.88	27.91	[1]19.91	19.94	18.29	17.99	.....	.....
	2.92	2.99	27.88	27.91	[1]19.97	19.99	18.20	17.87	.....	.....
	2.87	2.99	27.94	28.00	[2]19.90	19.99	18.15	18.15	.....	.....
	2.97	3.03	28.03	28.00	[2]19.90	19.92	18.15	17.92	.....	.....
	.....	.....	27.93	28.00	.....	.....	18.27	17.95	.....	.....
	.....	.....	27.77	28.00	.....	.....	18.28	17.95	.....	.....
	.....	.....	27.75	.....	.....	.....	18.08	17.95	.....	.....
	.....	.....	27.84	.....	.....	.....	18.19	.....	.....	.....
	.....	.....	.....	.....	.....	.....	18.15	.....	.....	.....

[1] Direct precipitation with magnesia mixture.

[2] Molybdate method.

TABLE I.—*Total phosphoric acid by gravimetric and volumetric methods, etc.*—Continued.

Analyst.	No. 1. Cottonseed meal.		No. 2. South Carolina phosphate.		No. 3. Disodium hydrogen phosphate.		No. 4. European slag.		No. 5. American slag.	
	Gravimetric method.	Volumetric method.	Gravimetric method.	Volumetric method.	Gravimetric method.	Volumetric method.	Gravimetric method.	Volumetric method.	Gravimetric method.	Volumetric method.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
A. D. Cook, N. Y. Exp.	3.01	3.08	28.52	28.60	20.51	20.60	19.08	18.83	.....	.....
Station, Geneva.	3.01	3.10	28.50	28.60	20.54	20.50	19.06	18.75	.....	.....
W. H. Andrews, N. Y.	3.01	3.01	28.58	28.55	20.31	20.40	18.72	18.60	.....	.....
Exp. Station, Geneva.	3.01	.....	28.64	28.60	20.38	20.45	18.75	18.75	.....	.....
	3.05	3.03	28.67	28.65	20.70	20.50	18.77	18.80	.....	.....
J. P. Street, N. J. Exp.	3.12	3.06	28.53	28.47	19.91	19.96	18.40	18.38	.....	.....
Station.	3.13	3.08	28.55	28.50	19.94	20.21	18.43	18.61	.....	.....
	3.17	3.12	.....	.....	20.01	.....	18.59	18.54	.....	.....
	.....	3.13	.....	.....	20.10	.....	.....	.....	.....	.....
W. Skinner, Md. Agr.	3.11	3.11	28.04	28.02	19.84	19.80	18.26	18.52	.....	.....
College.	3.03	3.05	28.01	28.03	20.01	19.89	18.38	18.62	.....	.....
J. R. Laughlin, Md. Agr.	3.00	3.22	27.91	27.93	19.95	19.80	18.28	18.57	.....	.....
College.	3.01	3.09	28.04	28.08	19.96	19.76	18.34	18.72	.....	.....
C. H. Jones, Vt. Exp.	3.10	2.93	28.58	28.50	20.54	20.50	18.46	18.42	18.10	17.98
Station.	3.12	2.95	28.62	28.45	20.66	20.60	18.32	18.45	18.16	17.95
	3.01	2.98	28.46	28.55	20.54	20.60	18.42	18.50	18.00	18.00
	.....	.....	28.50	.....	.....	.....	18.36	.....	18.10	.....
B. O. White, Vt. Exp.	3.10	.....	28.66	.....	20.64	.....	18.48	.....	18.06	.....
Station.	.....	.....	28.52	.....	.....	.....	18.42	.....	18.00	.....
	.....	.....	28.48	.....	.....	.....	.....	.....	.....	.....
F. S. Shiver, S. C. Agr.	3.07	3.02	28.24	28.15	20.14	19.97	18.42	18.20	17.95	17.73
College.										
John Thompson, S. C.	3.09	3.09	28.23	28.45	20.07	20.03	18.40	18.46	17.91	17.60
Agr. College.										
C. C. McDonnell, S. C.	3.06	3.08	28.25	28.39	20.05	20.08	18.38	18.41	17.88	17.76
Agr. College.										
C. Glaser, Baltimore	3.02	3.02	27.98	[1]27.76	19.89	[1]19.88	.....	.....	.....	.....
	.....	[2]3.24	.....	[2]26.53	19.85	[2]20.54	.....	.....	.....	.....
J. L. Stinson, Miss. Agr.	2.99	.....	27.87	.....	19.85	.....	18.03	.....	.....	.....
College.	3.02	.....	27.85	.....	19.93	.....	18.00	.....	.....	.....
	3.03	.....	.....	.....	.....	.....	18.01	.....	.....	.....
	.....	.....	.....	.....	.....	.....	18.11	.....	.....	.....
W. R. Perkins, Miss.	3.12	.....	28.11	.....	.....	.....	18.41	.....	17.69	.....
Exp. Station.	3.17	.....	28.09	.....	.....	.....	18.43	.....	17.64	.....
J. S. Carroll, Miss. Agr.	3.11	3.09	28.02	28.00	19.88	19.90	18.43	18.27	17.88	17.77
College.	3.11	3.10	27.99	28.00	20.01	20.00	18.31	18.22	17.82	17.85
	.....	3.06	.....	28.00	.....	19.92	.....	18.25	17.91	17.75
	.....	3.07	.....	28.05	.....	19.95	.....	18.25	17.82	17.70
	.....	3.12	.....	.....	.....	.....	.....	18.20	.....	.....
	.....	3.12	.....	28.10	.....	20.05	.....	18.15	.....	.....
E. W. Magruder, Miss.	3.01	3.10	28.04	28.00	19.94	19.85	18.42	18.30	17.82	17.75
Agr. College.	2.99	3.09	28.06	27.95	19.96	19.90	18.45	18.32	17.85	17.70
	.....	3.12	27.97	28.05	.....	19.90	18.38	18.25	17.77	17.65
	.....	3.11	28.09	27.95	.....	20.00	18.54	18.25	17.76	17.68
	.....	3.12	27.97	.....	.....	19.92	18.51	18.27	.....	.....
	.....	.....	28.08	.....	.....	19.95	18.55	18.30	.....	.....

[1] Washed with hot water. [2] Washed with cold water.

TABLE I.—*Total phosphoric acid by gravimetric and volumetric methods, etc.*—Continued.

Analyst.	No. 1. Cottonseed meal.		No. 2. South Carolina phosphate.		No. 3. Disodium hydrogen phosphate.		No. 4. European slag.		No. 5. American slag.	
	Gravimetric method.	Volumetric method.	Gravimetric method.	Volumetric method.	Gravimetric method.	Volumetric method.	Gravimetric method.	Volumetric method.	Gravimetric method.	Volumetric method.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
E. B. Ferris, Miss. Exp.	.....	3.07	28.01	28.10	19.92	19.90	18.74	18.35	18.14	17.60
Station.	.....	3.07	.....	28.10	19.93	.....	18.68	18.25	18.19	17.60
	.....	.....	.....	28.20	.....	.....	.....	.....	.....	.....
B. W. Kilgore, Miss.	3.04	3.09	27.95	28.10	19.92	19.85	[1]18.17	18.10	17.80	17.65
Agr. College.	.....	3.10	28.05	28.10	19.88	19.85	[1]18.14	18.15	17.85	17.70
	.....	.....	.....	.....	.....	19.92	[1]18.10	18.17	17.85	17.65
	.....	.....	.....	.....	.....	19.92	[2]18.52	18.25	.....	17.65
	.....	.....	.....	.....	.....	19.85	[2]18.54	18.10	.....	17.65
C. C. Moore, U. S. Dept. of Agr.	.....	3.02	.....	27.45	.....	19.72	.....	18.04	.....	17.47
E. G. Runyan, U.S. Dept. of Agr.	.....	.....	.....	.....	.....	.....	18.12	.....	17.48	.....
C. B. Williams, N. C.	3.02	.....	27.93	.....	19.89	.....	18.19	.....	17.27	.....
Exp. Station.	3.03	.....	27.95	.....	19.79	.....	18.09	.....	17.42	.....
	2.98	.....	27.86	.....	19.77	.....	18.11	.....	.....	.....
F. G. Kelly, N. C. Exp.	2.97	.....	27.72	.....	19.85	.....	18.17	.....	.....	.....
Station.	2.98	.....	27.72	.....	.....	.....	18.10	.....	.....	.....

[1] One precipitation.

[2] Precipitated twice to get rid of iron.

Volumetric determination of phosphoric acid in referee's samples by C. C. Moore.

Sample No.	Number determinations.	Maximum.	Minimum.	Average.
		Per cent.	*Per cent.*	*Per cent.*
1	10	3.03	3.01	3.02
2	10	27.61	27.32	27.45
3	40	20.05	19.50	19.72
4	10	18.24	17.98	18.04
5	10	17.54	17.36	17.47

DISCUSSION OF RESULTS.

The results of No. 1, cottonseed meal, which contains about 3 per cent of phosphoric anhydrid, are good by both methods, with the exception of two. This is especially true, when the results of each worker by the two methods—presumably on the same solution in most cases—are compared with each other. No one has found difficulty with the volumetric method on this sample, and as this fairly represents the percentages to be determined in insoluble work in fertilizers it seems fair to assume that the method has reached the stage where it is safe and reliable for this class of work at any rate.

On sample No. 2, ground South Carolina rock, which, we think, contains about 28 per cent of phosphoric anhydrid, while the results of different analysts by the same methods vary considerably, the results of the same analyst by the two methods agree within a fairly reasonable error of analysis. Workers who have gotten high results

by one method have gotten high results by the other, and vice versa. This indicates that the differences belong not to the methods themselves but to the analyst, and might reasonably be ascribed to differences in solutions, to the aliquots taken, etc.

When the high percentage of phosphoric acid in this sample is considered and the work of the past taken as a guide, the results are as good as would be expected.

The results on sample No. 3, disodium hydrogen phosphate (containing 19.83 per cent phosphoric anhydrid), compare well with each other when the results of the same analyst are considered. Quite a number of the workers have gotten high results on this sample.

Samples 4 and 6 (European and American slag meals, respectively) contain considerable iron. The volumetric results on these samples are, as a rule, lower than the gravimetric ones and, we think, more nearly represent the amount of phosphoric acid in these samples on account of the contamination of the "white precipitate" with iron from materials containing large amounts of this substance. All the "white precipitates" in my work with these samples were shown by test to contain iron, and the other workers in this laboratory found the same to be true. This is nothing new, however. The volumetric method would then seem to be a very desirable one where much iron and aluminum are present in the materials to be analyzed. It especially commends itself for use on soils, slags, etc.

Judging from the results presented, the volumetric method shows itself to be a good and reliable one. The comments of four of the twenty-two analysts taking part in the work show that they do not have it sufficiently well in hand to get thoroughly trustworthy results in all cases. The other workers have had no difficulty with it. It appears that those who have used the method sufficiently to become thoroughly familiar with its workings get good and reliable results and those who have used it most like it best.

COMMENTS OF ANALYSTS.

W. L. Mitchell, Connecticut.—The results by the volumetric method are, on the whole, very satisfactory. This good success may be attributed in part to the form of Hirsch funnel used. Those employed in past years did not contain perforations enough and were not of sufficient capacity. A more suitable form was used in this year's work. Precipitation was made at 65°, and filtration on Hirsch funnel with asbestos filter. Cold water was used for washing.

W. Skinner, Maryland.—Had difficulty in determining end reaction with Nos. 2 and 4, as the solution in the alkali became intensely blue. No. 2 is very high in aluminum, while No. 4 is high in iron. It was found difficult to wash No. 4 free from acid, which may account for high volumetric results on this sample. I also had considerable difficulty in getting concordant gravimetric results on this sample.

J. R. Laughlin, Maryland.—Also had difficulty in getting concordant results on No. 4.

C. H. Jones, Vermont.—States that he uses asbestos filter and does not have the trouble with the end reaction he formerly did when using paper. He further states that he compared the volumetric and gravimetric methods on 30 fertilizers with close agreement of results. The solutions were made in sulphuric acid. In one case, where considerable organic matter was present, the results did not agree well, but the trouble was traced to the solution, and he thinks comparative determinations on the same solutions should be made before deciding a method to be at fault.

F. S. Shiver, South Carolina.—Precipitated at room temperature and allowed to stand over night. Then filtered and washed with cold water till free of acid.

C. C. McDonnell and John Thompson, South Carolina.—Both state that they could not get uniform results with the volumetric method. The ones reported are the averages of results showing closest agreement.

C. Glaser, Baltimore, Md.—Used warm water (80° to 90°) for washing, and found it worked better than cold water. He washed with about 300 cc.

C. C. Moore, United States Department of Agriculture.—States: I can detect no difference in the two methods of washing and found no advantage in the use of nitric acid. I generally use the perforated platinum cone with filter paper with slight suction. The washing is continued until the funnel three times filled does not neutralize one-half drop of the standard alkali.

The alkaline solution was standardized by actual determinations of the chemically pure salt, No. 3. Ten determinations, using from 0.01 to 0.12 gram of the salt, were made. The end reactions were very uniform. There was but slight variation, and the maximum and minimum samples gave identical results.

J. S. Carroll, Mississippi.—It was found necessary to precipitate twice with molybdic solution to get rid of iron in samples 4 and 5. The "white precipitates" were then not entirely free from it.

Filtration through an asbestos filter on a perforated disk in a Gooch crucible tube was found to be the most satisfactory way. The glazed porcelain Gooch crucible with false bottom covered with an asbestos filter also works well.

TEST OF METHODS OF PRECIPITATION WITH VOLUMETRIC METHOD.

Considerable time was given in the laboratory of the Mississippi Agricultural College to testing methods of precipitation, the quantity of molybdic solution best to use in the volumetric method, etc. These investigations were made on the cottonseed meal, disodium hydrogen phosphate, and European slag samples sent out by the referee. The work was done by Messrs. E. W. Magruder, J. S. Carroll, and the referee. The results are presented in Table II, which follows:

TABLE II.—*Test of methods of precipitation when using the volumetric method.*

COTTONSEED MEAL.

Sample.	Gravimetric results by usual method.	Volumetric results.	Time allowed for precipitation.	Temperature of precipitation.	Amount of substance used.	Amount molybdic solution used.
	Per cent.	*Per cent.*	*Hours.*	°*C.*	*Grams.*	*cc.*
No. 1	3.10	3.04, 3.04, 3.03, 3.05	½	65	0.4	15
		3.05, 3.05	½	65	.4	20
		3.12, 3.16, 3.17, 3.09, 3.10, 3.17, 3.07.	½	65	.4	30
		2.98, 3.00, 2.98, 2.91, 2.75	1	30	.4	15
		2.93, 3.08, 3.03, 3.06	1	30	.4	30
		3.04, 3.04, 3.03, 3.02, 3.05, 3.04	7	31–27	.4	20
No. 1 *a*	.52	0.54, 0.55, 0.54, 0.54, 0.54, 0.53	17	31–27	.4	20
No. 1 *b*	.26	0.27, 0.26, 0.27, 0.27, 0.26, 0.26	17	31–27	.4	20
Reverse precipitation		3.07, 2.91, 2.90	1	30	.4	15
		3.10, 3.10, 3.07	1	30	.4	30

DISODIUM HYDROGEN PHOSPHATE.

No. 3	19.95	19.85, 19.90, 19.90, 19.85, 19.85.	½	65	0.2	30
		20.00, 20.00, 19.95, 19.97, 19.92, 19 95, 19.90, 19.92, 20.05, 19.97.	½	65	.2	35
		20.10, 20.25, 20.20, 20.20, 20.18, 20.15, 20.22, 20.20.	½	65	.2	50
		19.84, 19.90, 20.00, 20.00, 19.50, 19.10.	½	65	.1	15
		20.25, 20.30, 20.25	½	65	.1	30
		20.20, 20.15, 20.40	¾	65	.1	30

TABLE II.—*Test of methods of precipitation when using the volumetric method*—Continued.

DISODIUM HYDROGEN PHOSPHATE—Continued.

Sample.	Gravimetric results by usual method.	Volumetric results.	Time allowed for precipitation.	Temperature of precipitation.	Amount of substance used.	Amount molybdic solution used.
	Per cent.	*Per cent.*	*Hours.*	°*C.*	*Grams.*	*cc*
		19.05, 19.42, 19.62	¾	31	.2	30
		19.95, 19.95, 20.00	¾	31	.2	50
		19.95, 19.95, 19.95	1	28	.2	35
		19.95, 19.90, 19.90	1	31	.2	40
		19.40, 19.30, 19.00, 19.90, 19.68, 19.50.	1	20	.2	32
		19.88, 19.85, 19.85	15.5	24–25	.2	32
		19.90, 19.89, 19.90	15.5	24–25	.2	55
Reverse precipitation		19.20, 19.30	½	20	.2	32
		19.95, 19.90, 19.90	1	20	.2	32
		19.95, 19.95, 19.98	1	28	.2	35
		19.95, 19.98, 19.95	1	31	.2	40
		20.05, 19.95, 19.90, 19.88, 19.83, 19.83.	½	30	.2	30
		20.05, 20.02, 19.98, 20.05, 19.98, 19.98.	½	30	.2	50
		EUROPEAN THOMAS SLAG.				
No. 4 (regular precipitation).	18.47	18.20, 18.23, 18.23	17	24–25	0.2	32
		18.25, 18.27, 18.27	17	24–25	.2	35
		18.33, 18.32, 18.25, 18.27	½	65	.2	40

From the results presented and observations made in the course of the work we feel warranted in drawing the following conclusions:

That at a temperature of 65° 10 cc of molybdic solution did not always precipitate the phosphoric acid in the cottonseed meal, containing about 3 per cent, in one-half hour, but 15 cc did, and as much as 25 cc of the molybdic solution could be added without giving high results. Thirty cc gave slightly but not very materially higher results than from 15 to 25 cc of molybdic solution. When precipitation was made at room temperature in the summer it could not be relied upon to be complete in one hour, but on standing over night this 3 per cent sample and ones containing 0.50 per cent and 0.25 per cent were complete in all cases. Large excess of molybdic solution did not give high results on these percentages when precipitation was made in the cold. An aliquot corresponding to 0.4 gram substance was used on these percentages.

For samples 3 and 4, containing 18 and 20 per cent phosphoric anhydrid, 30 cc of molybdic solution gave complete precipitation at 65°, and 35 and 40 cc could be used without increasing the results, but 50 and 55 cc of solution gave high results, showing that a large excess of molybdic solution in the volumetric method will give high results when precipitation is made at 65°. One hour in the cold with the above amounts of molybdic solution did not always give complete precipitation, but on standing over night (from fifteen to seventeen hours) all the phosphoric acid came down, and a large excess of molybdic solution (45 to 55 cc) did not give as high results as when precipitation was made at 65°. Precipitation in the cold took place quicker with these high percentages than with the low ones.

REVERSE PRECIPITATION.

The addition of the phosphate solution to the molybdic solution containing the necessary ammonium nitrate brought down the ammonium phosphomolybdate somewhat quicker than the usual procedure, but the difference did not seem to be very great.

PRECIPITATION BY SHAKING IN THE COLD.

The following tests were carried out to see if shaking at room temperature would give complete precipitation. The machine used was the revolving one commonly employed in determining the available phosphoric acid in slags by the Wagner citrate method. Shaking was continued for from fifteen to sixty minutes, the machine being run by a small gas engine at the rate of about sixty revolutions per minute.

The results show precipitation to have been complete in all these 73 determinations, though the time of shaking was only fifteen minutes in a number of the tests and filtration was carried out at once. A small excess of molybdic solution brought down all the phosphoric acid in this work, and the use of a double quantity did not increase the results as it did when precipitation was made at the usual temperature, 65°.

It appears on the basis of these results that this means of precipitation by shaking at room temperature in some convenient form of machine is a most promising one for giving quick and complete precipitation, and the conditions in it are not favorable for the precipitation of molybdic acid along with the ammonium phosphomolybdate. It is recommended for further study.

TABLE III.—*Test of shaking as a means of precipitation of phosphoric acid in the volumetric method.*

[E. W. Magruder, analyst, Mississippi Agricultural College.]

Sample.	Gravimetric results by usual method.	Volumetric results by shaking.	Time of shaking.	Temperature of precipitation	Amount of substance used.	Amount of molybdic solution used.
	Per cent.	*Per cent.*	*Minutes.*	*°C.*	*Grams.*	*cc.*
COTTONSEED MEAL.						
No. 1	3.10	3.00, 3.03, 3.03	60	31	0.4	20
		3.06, 3.03, 3.03	30	31	.4	20
No. 1*a*	.52	0.55, 0.55, 0.55, 0.55, 0.53	30	25	.4	15
		0.55, 0.56, 0.55, 0.55, 0.56, 0.55	30	25	.4	30
No. 1*b*	.26	0.31, 0.30, 0.30, 0.27, 0.27, 0.28	30	27	.4	15
		0.26, 0.26, 0.27, 0.26, 0.27, 0.27	30	27	.4	30
PHOSPHATE ROCK.						
No. 2	28.04	28.03, 28.05	30	24	0.1	25
		28.05, 28.08	30	24	.1	50
DISODIUM HYDROGEN PHOSPHATE.						
No. 3	19.95	20.00, 19.98, 20.00	30	27	0.2	40
		19.92, 19.95	40	30	.2	32
		19.95, 19.90, 19.88	20	28	.2	32
		19.90, 19.75, 19.85	20	28	.2	55
		19.95, 19.95, 19.92	15	28	.2	32
		19.92, 19.90, 19.90	15	28	.2	55

TABLE III.—*Test of shaking as a means of precipitation of phosphoric acid in the volumetric method*—Continued.

EUROPEAN SLAG.

Sample.	Gravimetric results by usual method.	Volumetric results by shaking.	Time of shaking.	Temperature of precipitation.	Amount of substance used.	Amount of molybdic solution used.
	Per cent.	*Per cent.*	*Minutes.*	*°C.*	*Grams.*	*cc.*
No. 4	18.47	18.25, 18.20, 18.20, 18.25, 18.20, 18.25, 18.25.	30	25	0.2	30
		18.20, 18.20, 18.25, 18.20, 18.30	30	25	.2	50

AMERICAN SLAG.

No. 6	17.80	17.65, 17.65, 17.65, 17.60, 17.67	30	24	0.2	30
		17.73, 17.70, 17.70	30	24	.2	50

FERTILIZERS.

Total	12.66	12.58	30		0.2	30
Soluble	9.00	8.97	30		.2	25
Insoluble	2.08	2.10	30		.4	20

RESULTS FROM MISCELLANEOUS FERTILIZER SAMPLES BY GRAVIMETRIC AND VOLUMETRIC METHODS.

Mr. F. S. Shiver, of the South Carolina Agricultural College, compared the gravimetric and volumetric methods on totals, solubles, and insolubles in a number of fertilizers. He precipitated at room temperature, allowed to stand over night, and washed with cold water.

Mr. E. W. Magruder also made a number of comparative tests in the course of regular work. Precipitation was made at 65° C. and allowed to stand fifteen minutes. The washing was done with cold water. Results are presented in Table IV.

TABLE IV.—*Results from miscellaneous fertilizers by gravimetric and volumetric methods.*

[F. S. Shiver, analyst, South Carolina Agricultural College.]

TOTALS.

Sample No.	Gravimetric method.	Volumetric method.	Sample No.	Gravimetric method.	Volumetric method.
	Per cent.	*Per cent.*		*Per cent.*	*Per cent.*
1	15.91	15.92	6	2.62	2.62
2	15.68	15.79			2.54
3	10.06	10.17	7	30.16	30.25
4	27.96	28.13	8	42.35	42.10
		28.18	9	15.91	15.96
		28.18			16.09
5	22.60	22.58	Average	20.36	20.40
		22.58			

TABLE IV.—*Results from miscellaneous fertilizers by gravimetric and volumetric methods*—Continued.

SOLUBLES.

Sample No.	Gravimetric method.	Volumetric method.	Sample No.	Gravimetric method.	Volumetric method.
	Per cent.	*Per cent.*		*Per cent.*	*Per cent.*
1	10.44	10.59	5	6.54	6.54
2	6.58	6.65			6.56
3	6.98	7.06	6	9.93	9.98
4	8.88	8.91	Average	8.23	8.29
		8.93			

INSOLUBLES.

1	1.32	1.24	8	1.09	1.07
2	1.27	1.11	9	1.47	1.46
3	1.50	1.46	10	1.34	1.38
4	1.36	1.32	11	1.23	1.23
5	1.83	1.77	12	1.28	1.29
6	1.62	1.72	Average	1.54	1.51
7	3.11	3.10			

[E. W. Magruder, analyst, Mississippi Agricultural College.]

INSOLUBLES.

869	0.90	0.97	876	1.45	1.49
		.96			1.47
		1.00			1.47
870	.90	.95	877	1.11	1.15
		.96			1.14
		.96			1.02
871	.88	.97	880	2.55	2.57
		.95			2.52
		.95			2.54
872	1.20	1.24	882	1.25	1.31
		1.24			1.31
873	3.71	3.65			1.30
		3.74	856	2.39	2.42
874	2.64	2.60			2.37
		2.62			2.41
		2.65			2.40
875	1.87	1.82			
		1.82			
		1.85			

TOTALS.

850	9.74	9.82
		9.88

PRECIPITATION AND WASHING.

Precipitation.—Two methods of precipitation were outlined in the instructions for the work the past season. One was to heat to about 65° and the other to about 50° before adding the molybdic solution. Most of the analysts precipitated at 65°, and at least three worked at room temperature and allowed to stand over night, when precipitation was completed in all cases.

Washing.—Nearly all of the analysts washed with water only. A large number of tests in one laboratory showed the decantation washing with dilute nitric acid to have no advantage. Washing once or twice by decantation with water, and then on the filter with enough water to make with the decantation washings about 200 cc. was found sufficient to free the precipitate of acid. In this laboratory we have not found it necessary to test the filtrate for acid after washing in this way, but proceed at once with the titration, a large number of tests having shown us that all acid has been removed. The precipitate may be washed with 300 cc. and in some cases as much as 400 or 500 cc of water without material loss, but it is to be remembered that ammonium phosphomolybdate is an acid salt which is slightly soluble in water, enough so to make the filtrate react acid to delicate indicators. Besides the danger of dissolving a small amount of the salt when very large amounts of water are used, some of the precipitate is liable to run through the filter when as much as 500 or 600 cc of water are used for washing.

FILTERS.

A number of forms of filters have been used with the volumetric method. Among these we would name in the order of preference: (1) That made by putting a perforated porcelain or other disk well filled with holes in the glass carbon filters used for Gooch crucibles, attaching a stiff piece of wire to the plate with which to push it out of the tube, and covering the plate with an asbestos filter. (2) The porcelain Gooch crucible, of good size, with false bottom well filled with holes, the bottom being covered with an asbestos filter. (3) The Hirsch funnel, with bottom well filled with holes and covered with an asbestos filter. (4) Funnel and filter paper with or without pressure.

STANDARD SOLUTION AND INDICATOR.

As the titration is to be made with phenolphthalein as indicator the solutions should be standardized with this indicator. We use alcohol distilled over sodium or potassium hydroxid (free from carbonates) for making the standard alkali. For each liter of solution dissolve the alkali in 120 to 130 cc of alcohol, filter from insoluble carbonates, and dilute with water. Much more alcohol than this should not be used, as it might give reduction and consequent coloration in the titration.

CARBON DIOXID IN DISTILLED WATER.

Distilled water obtained from water containing large amounts of bicarbonates is liable to have considerable carbon dioxid in solution. There is no objection to the use of this water for washing the yellow precipitate, but in transferring the precipitate to the beaker or precipitating vessel and diluting in the process of titration a small error may be introduced. Boiling the water used for this purpose obviates any difficulty from that source.

RECOMMENDATIONS.

(1) That precipitation at room temperature in the volumetric method by means of shaking in some convenient form of shaking machine be made a subject of study.

(2) That the volumetric method for the estimation of phosphoric acid by titrating the ammonium phosphomolybdate precipitate with standard alkali be made an optional method.

DESCRIPTION OF VOLUMETRIC METHOD.

Dissolve substance according to methods (A2), (A5), (A6), or (A7) (Bulletin 46, Division of Chemistry, United States Department of Agriculture, p. 12), preferably by (A5) when these acids are a suitable solvent, and dilute to 200 cc with water. For percentages below 5, measure out aliquot corresponding to 0.4 gram substance, for percentages between 5 and 20 use aliquot corresponding to 0.2 gram substance, and for percentages above 20 use aliquot corresponding to 0.1 gram substance. Add 5 to 10 cc nitric acid (or the equivalent in ammonium nitrate), depending on the method of solution, nearly neutralize with ammonia, dilute to from 75 to 100 cc, heat in water bath to from 60 to 65°, and for percentages below 5 add from 20 to 25 cc freshly filtered molybdic solution (official), for percentages between 5 and 20 add from 30 to 35 cc solution, and for percentages above about 20 add from 30 to 35 cc molybdic solution. Stir, let stand about fifteen minutes, filter *at once*, wash once or twice by decantation with water, using from 25 to 30 cc each time, agitating the precipitate thoroughly and allowing to settle, transfer to filter and wash five or six times, using enough water to make with the decantation washings about 200 cc. Transfer precipitate and filter to beaker or precipitating vessel, dissolve in small excess of standard alkali, add a few drops of phenolphthalein solution and titrate back with standard acid (nitric).

PREPARATION OF REAGENTS.

(*a*) *Standard potassium hydroxid solution.*—This solution contains 18.17106 grams of potassium hydroxid to the liter. It is prepared by diluting to 1 liter 323.81 cc of normal potassium hydroxid, which has been freed from carbonates. One hundred cc of the solution should neutralize 32.38 cc normal acid. One cc is equal to one mg P_2O_5 (1 per cent P_2O_5 on basis of 0.1 gram substance).

(*b*) *Standard nitric acid solution.*—The strength of this solution is the same as, or one-half that of, the standard alkali solution, and is determined by titrating against that solution, using phenolphthalein as indicator.

(*c*) *Phenolphthalein solution.*—One gram of the phenolphthalein is dissolved in 100 cc of alcohol.

In addition to the method as adopted the following suggestions are offered by the referee:

Filters in the following order of preference are recommended: (1) That made by putting a perforated porcelain or other disk, well filled with holes, in the glass carbon filters used for a gooch, attaching a stiff piece of wire to the plate with which to push it out of the tube, and covering the plate with an asbestos filter. (2) The porcelain gooch of good size with false bottom well filled with holes, the bottom being covered with an asbestos filter. (3) The Hirsch funnel with bottom well filled with holes and covered with an asbestos filter. (4) Funnel and filter paper with or without pressure.

Instead of precipitating at 65° C, precipitation may be made at room temperature by shaking for about thirty minutes in some convenient form of shaking machine.

The president called for papers on phosphoric acid and discussion of the report of the referee.

In regard to the estimation of available phosphoric acid in basic slag, Mr. Wheeler stated that he had attended the meeting of the representatives of the German experiment stations last summer, and that in view of the fact that much difficulty has been experienced in the past in obtaining comparative results even on the same sample in different German laboratories, Professor Wagner proposed that a 2 per cent

citric acid solution be adopted in lieu of the acid citrate solution previously employed, and that the conditions of treatment should remain the same as heretofore. His suggestion was very strongly supported by Professor Maercker, of Halle, and a few others. The result is that members of the association are to make a trial of the method, and they are to meet, probably in January, in Berlin, in order to consider its final adoption. It is supposed to give about 1 per cent more of available phosphoric acid in ordinary slags than the usual citrate method, but it is hoped that it will accord very closely with the results obtained in vegetation experiments, and such further experiments will be conducted in order to ascertain if it be more reliable as a guide to the assimilability than the method formerly employed.

At the conclusion of the discussion, the committee appointed to wait upon the Secretary and Assistant Secretary of Agriculture announced that it had performed the duties imposed upon it. The Secretary of Agriculture received the committee and promised to attend the convention at some period of its sessions, but was unable to name a definite hour. The Assistant Secretary was absent from the city and therefore could not be seen.

The president called for the reports of the committee on recommendations of referees. The committee made the following report upon the recommendation of the referee on nitrogen:

The committee upon recommendations of referees recommends the adoption of the recommendation of the referee on nitrogen that a further trial be made of the potassium permanganate methods for the determination of the availability of organic nitrogen in fertilizers.

The report of the committee was adopted.

The committee also presented the following report on the recommendations of the referee on potash:

Your committee concurs with the referee in the recommendation that the Lindo-Gladding method be adopted for determining water soluble potash in wood ashes and cotton-hull ashes, special attention being paid to the words "The precipitate should be perfectly soluble in water," found in the description of this method on page 2, Circular No. 4, United States Department of Agriculture, Division of Chemistry.

The report of the committee was approved.

The committee also presented the following report on the recommendations of the referee on phosphoric acid:

Your committee recommends that the volumetric method for the determination of phosphoric acid as described in the report of the referee be adopted as an optional method.

After a somewhat prolonged discussion the action of the committee was approved.

At 4 o'clock the association adjourned until Saturday morning.

SECOND DAY.

SATURDAY—MORNING SESSION.

After the dispatch of some routine business, and announcements by the secretary and others, the president called for the report of the referee on soils and ash. The report was read by the referee, Mr. Harry Snyder.

REPORT ON SOILS AND ASH.

By Harry Snyder, *Referee.*

The referees on soils and ash were instructed by this association to continue the investigations relating to the use of a fifth normal hydrochloric acid solution as a solvent for the available phosphoric acid of the soil, and the calcium chlorid and the ammonium chlorid methods for the determination of available potash.

It was also considered advisable to include the determination of (1) the total nitrogen, (2) the nitrogen content of the humus, and (3) the calcium in the form of calcium carbonate. A circular letter was issued inviting the cooperation of all station chemists. Fourteen favorable replies were received, and reports were obtained from six laboratories.

It was thought best to secure results from as large a number of soils as possible, so that the accuracy of the methods might be tested on different types of soil. Ten soils were selected for the work. Some difficulty was experienced in securing suitable samples. While it is claimed that $60,000,000 worth of commercial fertilizers are annually used in this country, it is surprising to find how small a number of soils are known from carefully conducted experiments to be deficient in one or more of the essential elements of plant food.

DESCRIPTION OF SOILS.

Soil No. 1. Deficient in available phosphoric acid.—This soil was secured by Mr. A. M. Peter, of the Kentucky Station, from the farm of Mr. Thomas R. Walker at Junction City, Ky. Experiments have shown that this soil is deficient in available P_2O_5, and is apparently well supplied with available potash, although there is no superabundance of that element. (Ky. Bulletin 61, pp. 11–14). This land had a few months previously received a dressing of 200 pounds per acre of acid phosphate.

Soil No. 2. Deficient in available potash.—From the Kentucky Agricultural Experiment Station. This soil has never received any fertilizer, and experiments conducted for a series of years indicate poverty in K_2O.

Soil No. 3. Deficient in available nitrogen.—From near Raleigh, N. C., furnished by Mr. Withers, of the North Carolina Experiment Station. This soil is deficient to a less extent in available P_2O_5.

Soil No. 4. Deficient in available nitrogen.—From the North Louisiana Experiment Station, furnished by Director Stubbs. This soil is also slightly deficient in available phosphoric acid, and while analyses have shown it to be deficient in potash we fail to see any response to any form of this ingredient. This soil has never been fertilized.

Soil No. 5. Mixed soils.—From the Ohio Experiment Station. This soil is now under investigation, and Director Thorne states that he is not yet prepared to say just what constituents are lacking. This sample was made up from twenty-seven separate samples taken from nine plots. Inasmuch as this soil is to be subjected to careful field tests with fertilizers, it will be interesting to observe to what extent the laboratory results agree with the field tests.

Soil No. 6. "Worn-out soil."—From near Columbia, Mo. Director Waters writes that this soil has been cropped without manures for over forty years, and it has been so reduced in crop-producing power that it yields less than 5 bushels of corn per acre, while adjoining land, as soil 6A, which has been properly manured, yields 60 bushels per acre.

Soil No. 7. Deficient in lime.—From the Rhode Island Experiment Station.

Soil No. 8. Virgin soil of average fertility.—From the Minnesota Experiment Station. Similar soils which have been cropped for thirty years show no material response when commercial fertilizers are applied, but are benefited by applications of stable manure.

Soil No. 9. Virgin soil of high fertility.—From the substation at Crookston, Minn. Similar soils which have been cropped for ten years are not benefited by applications of commercial fertilizers, but, if anything, show a slight decline in yields when they are used.

Soil No. 10.—For mechanical analysis.

DIRECTIONS FOR WORK ON A. O. A. C. SOIL SAMPLES FOR 1898.

(1) The Determination of Moisture.

Use the official method described in Bulletin 46, page 38, Division of Chemistry, United States Department of Agriculture. Calculate all results to the water-free basis.

(2) The Determination of Phosphoric Acid Soluble in N/5 HCl.

(*a*) *Preliminary treatment.*—Digest 20 grams of soil with 200 cc of N/5 HCl in a water bath at 40° for five hours. Titrate 20 cc of the clear filtrate against a standard KOH solution, using phenolphthalein for the indicator. From the data thus secured calculate the amount of HCl necessary to be added so that the solution will be N/5 after allowing for the acid neutralized.

(*b*) *The determination.*—Place from 50 to 100 grams of soil in an Erlenmeyer flask, and add 10 cc of acid corrected for neutralization, as directed under (*a*), for every gram of soil used. The flask is corked with a rubber stopper which carries a thermometer. The flask is placed in a water bath previously heated to 40°. The soil is digested for five hours at a temperature of 40°, and the contents of the flask are thoroughly shaken every half hour during the digestion.

The solution is filtered through a ribbed filter of two thicknesses of paper, refiltering the first portion if cloudy. The filter should be large enough to receive the entire contents of the flask. Before filtering the contents of the flask are well shaken. From 400 to 600 cc of the filtrate (at 20°) are evaporated to dryness after adding from 1 to 3 cc HNO_3. If there be any appreciable amount of organic matter present, the residue is to be carefully ignited. Moisten the residue with HCl and add from 50 to 100 cc distilled water and digest. Filter, neutralize with ammonia, add 5 cc strong HNO_3 and 15 grams of ammonium nitrate in solution. Complete the determination according to one of the official methods given for the determination of phosphoric acid, or use the Goss method, as given in Circular 4 to accompany Bulletin No. 46.

(3) The Determination of Potash Soluble in N/5 $CaCl_2$.

Prepare a N/5 solution of $CaCl_2$ by dissolving the chemically pure salt in distilled water and titrating against standard $AgNO_3$, using potassium chromate as an indicator.

Digest 30 grams of soil with 500 cc of this solvent for five hours at 40°, as directed in section 2. Evaporate 400 cc of the filtrate to dryness in a porcelain dish after adding a few cc of HCl and HNO_3. Take up with water, add ammonium hydrate to alkaline reaction, and precipitate lime, etc., with ammonium carbonate and oxalate. Filter, add ½ cc of concentrated H_2SO_4 to the filtrate and evaporate to dryness in a large platinum dish. Thoroughly dry in the air bath at from 140° to 150°, ignite, and continue the determination by the Lindo-Gladding method.

(4) The Determination of Potash Soluble in Ammonium Chlorid.

Make up a N/20 solution of ammonium hydrate by titrating against standard acid, using cochineal as the indicator, and dissolve 10.68 grams of chemically pure crystalline NH_4Cl in each liter of the same.

Digest 50 grams of soil with 500 cc of this solvent exactly as under 2. Evaporate 400 cc of the filtrate to a small volume in a porcelain dish, transfer to a good-sized platinum dish, add ½ cc of concentrated H_2SO_4, and finish the evaporation over a water bath. Heat in the air bath at from 140° to 150°, ignite, and continue the determination by the Lindo-Gladding method.

(5) The Determination of Total Nitrogen.

Use the method outlined on page 2 of Circular No. 2 accompanying Bulletin No. 46. Nitrates are not to be included.

(6) The Nitrogen Content of the Humus.

Place from 10 to 25 grams of soil in a 200 cc glass-stoppered bottle. Add 150 cc of a 1 per cent solution of HCl; if the soil contain a higher per cent of lime use a 2 per cent HCl solution. Allow the acid to act on the soil for about five hours, with occasional shaking of the contents of the flask. Decant into a filter, add a fresh quantity of acid to the soil, decanting at the end of two hours. Continue to treat the soil with acid until no reaction for calcium is obtained; then wash with distilled water until the washings are only slightly turbid to $AgNO_3$. The soil on the filter is then transferred to the glass-stoppered bottle, using 150 cc of a 3 per cent NaOH solution. The bottle is occasionally shaken, and after from three to five hours the solution, after settling, is decanted into a 500 cc flask. A fresh quantity of alkali is added, and the treatment is continued until the extract becomes nearly colorless. Three or four treatments are usually necessary. The solution in the flask is made up to 500 cc, shaken well, allowed to settle, and then a 200 to 400 cc filtered portion is placed in a Kjeldahl digestion flask, neutralized with H_2SO_4 and evaporated nearly to dryness. The nitrogen determination is completed in the usual way.

(7) The Determination of Calcium Carbonate.

(*a*) To 100 grams of soil in a large flask or bottle add 2,500 cc of water saturated with pure CO_2 (made from Na_2CO_3 or $NaHCO_3$). Shake the flask twice a day and allow the solution to act on the soil for three days. To 2,000 cc of the clear filtrate add HCl until acid and evaporate to dryness. In the presence of organic matter the residue is carefully ignited and then dissolved in 50 cc distilled water and 2 cc HCl. Ammonia is added; any precipitate obtained is removed by filtering. Ammonium chlorid is added and the calcium is then precipitated with ammonium oxalate. The calcium is determined volumetrically, with a N/10 permanganate solution. Report results as CaO.

(8) Determination of Coarse and Medium Sand.

Place 10 grams of soil in a small beaker; add about 100 cc of water and heat for ten minutes on a sand bath. During the heating pestle the soil with rubber-tipped glass rod. Connect the 0.5 mm sieve by means of a Gooch crucible fitting with a

filtering flask. Pour the contents of the beaker into the sieve, using the glass rod if necessary to prevent clogging. Wash the material in the sieve with distilled water until the washings are clear, then pass 100 cc more water through the sieve. Remove the sieve to a drying oven, and when dry transfer the contents to a crucible. Ignite, and weigh.

The contents of the flask are then passed through the 0.25 mm sieve, and the particles between 0.5 and 0.25 mm determined.

Determine the moisture in a separate sample of soil, and report all results on dry soil.

The results obtained by different analysts in respect of phosphoric acid are found in the following table:

Soil No.	Description.	Total P_2O_5.	Solution HCl.	Per cent phosphoric acid soluble in N/5 HCl.									
				New Mexico.		South Carolina, Thompson.	Minnesota, Snyder.	Canada.		Rhode Island, Hartwell.	Massachusetts, Hasskins.	Department of Agriculture, Moore.	Average.
				Goss.	Holt.			Shutt.	Charran.				
		Per cent											
1	Deficient in P_2O_5	0.05	N/4.99	0.002	0.004	0.002	0.003	0.004	0.004	0.0025	0.0013	0.002	0.0026
4	Deficient in P_2O_5	.....	N/4.99	.....	.....	.....	.002	.0026	.....	.....	.....	.....	.0023
5	Deficiency unknown	.10	N/4.99	.....	.....	.....	.003	.....	.....	.....	.0024	.003	.0028
6	General poverty	.05	N/4.49	.002	.003	.002	.002	.....	.....	.....	.....	.....	.0022
8	Virgin soil, average fertility	.17	N/4.74	.022	.019	.033	.032	.....	.019	.0170	.0300	.038	.0260
9	Virgin soil, high fertility	.23	N/2.60	.063	.065	.061	.061	.035	.028	.....	.0670	.058	.0550

The HCl solution was N/5 after correcting for basic material in the soils

Soil No. 9 digested in N/5 HCl without correcting for basic material gave 0.044 per cent P_2O_5.

The phosphoric acid results are as a whole satisfactory. Soil No. 1, known to be deficient in available P_2O_5, shows an average of 0.0029 per cent soluble in fifth normal hydrochloric acid, or less than 100 pounds per acre. Similar results were obtained from soil No. 4, which is deficient in P_2O_5. In soil No. 6, the one of general poverty, there is only 0.0022 per cent soluble P_2O_5. Soil No. 5, according to the analytical results, is deficient in available P_2O_5. A recent bulletin from the Ohio Experiment Station states that field experiments "apparently justified the conclusion that, for the soils and crops under test, phosphoric acid is at present the most important constituent of a fertilizer, with nitrogen and potash following in the order named." In the case of this soil the laboratory results anticipated the field results. With soils Nos. 8 and 9, known to contain liberal amounts of phosphoric acid, the results show respectively ten and twenty times as much soluble phosphoric acid as in the case of soils deficient in this element.

The presence of two-thousandths of a per cent of an element in a soil report may seem to many like a very small amount, and to be within the limits of error in ordinary analytical work. The percentage amounts are small because such a large amount of soil is used, and not because there is only a small weighable amount of material. In fact, nearly 40 mg of magnesium pyrophosphate are secured for final weighing. In water analysis the figures for albuminoid ammonia would be even less than 0.002 per cent calculated on a percentage basis.

The citric acid method, as proposed by Dyer, has also been subjected to further investigation, with the following results:

Analyst.	Soil No. 1. Deficient in available P_2O_5.	Soil No. 4. Deficient in available P_2O_5.	Soil No. 8. Virgin soil, not deficient. Average fertility. P_2O_5.	Soil No. 9. Virgin soil, not deficient. High fertility. P_2O_5.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Shutt, Canada	0.0054	0.0026	0.0197	0.0189
Charran, Canada	.0054	.....	.....	.0196
Hummel, Minnesota	.0048	.....	.0210	.0175

While the results by citric acid show a difference between soils deficient and soils well stocked with available phosphoric acid, the method does not make as sharp a distinction as the N/5 HCl method. In fact, citric acid shows but little difference between the amounts of soluble phosphoric acid in soils 8 and 9, while the N/5 HCl shows a marked difference. Soil No. 9 is much more liberally supplied with available phosphoric acid. In former reports it has been shown that 1 per cent citric acid fails to give satisfactory results for the determination of the available P_2O_5 of the soil. (Goss, p. 87, Association Report, 1896.)

Some objection has been made against the use of N/5 HCl as a solvent for available phosphoric acid on the ground that it is too active a solvent. In the case of soil No. 1, deficient in available P_2O_5, citric acid was a more active solvent than N/5 HCl, while in the case of soil No. 9, which contains a liberal supply, the N/5 HCl was the more active solvent. This difference, however, is in favor of the HCl. How this would hold in the case of a large number of soils of course can not be told.

POTASH.

[Soluble in N/5 $CaCl_2$.]

Analyst.	Soil No. 2. Deficient in available K_2O.	Soil No. 8. Virgin soil. Average fertility. K_2O.	Soil No. 9. Virgin soil. High fertility. K_2O.
	Per cent.	*Per cent.*	*Per cent.*
C. C. Moore, U. S. Dept. Agr	0.0090	0.0170	0.0270
F. S. Shiver, South Carolina	.0030	.0106	.....
Jno. Thompson, South Carolina	.0027	.0095	.0295
B. L. Hartwell, Rhode Island	.0080	.0136	.....
Harry Snyder, Minnesota	.0070	.0156	.....

[Soluble in alkaline NH_4Cl.]

C. C. Moore, U. S. Dept. Agr	0.0090	.....	.....
F. S. Shiver, South Carolina	.0067	.....	.....
Jno. Thompson, South Carolina	.0071	.....	.....
B. L. Hartwell, Rhode Island	.0148	.....	.....
Harry Snyder, Minnesota[1]	.0142	0.02	0.077

[1] Huston reports on this same soil 0.0137. Bulletin No. 49, U. S. Dept. Agr.

Potash results from Minnesota Experiment Station Laboratory by the referee.

	Per cent.
Soil No. 1.—Fair amount of available K_2O	0.0092
	.0089
Soil No. 2.—Deficient in available K_2O	.013
	.0154
Soil No. 5.—Fair amount of available K_2O	.0123
	.0119
Soil No. 6.—General poverty	.019
	.023
Soil No. 8.—Virgin soil, average fertility	.02
Soil No. 9.—Virgin soil, high fertility	.077

In the above determinations the double salt of potassium and platinum was obtained in the usual way and was then dissolved in distilled water acidified, and the platinum determined by electrolysis. By means of electrolysis it is possible to make potassium determinations and avoid the foreign materials usually present in the final product.

In soil analysis it is quite essential that the impurities be removed, because they are present in larger amounts than in ordinary fertilizer work. The electrolytic method for the determination of potash is particularly applicable to soil work where abnormal amounts of impurities are present. The cause of the unusual discrepancies in the results on soil potash in former years has undoubtedly been due to the variable amounts of impurities present in the final product.

Soluble in 1 per cent citric acid.

[Results reported by Mr. Shutt, chemist, Dominion Experiment Farms.]

	Soil No. 1. Fair amount of available K_2O.	Soil No. 4. Fair amount of available K_2O.	Soil No. 8. Virgin soil, average.	Soil No. 9. Virgin soil, high.
K_2O solution, soluble in 1 per cent citric acid solution	*Per cent.* 0.0129	*Per cent.* 0.0126	*Per cent.* 0.0111	*Per cent.* 0.0204

In the case of the ammonium chlorid method for the determination of available potash, it is to be observed that a higher per cent of soluble potash was obtained from soil No. 1, deficient in available potash, than from No. 2, which contains a fair amount, and that soil No. 6, general poverty, shows more soluble potash than No. 8, a virgin soil of average fertility. The method gives valuable indications where the soils most deficient in potash are compared with those which are most liberally supplied with potash, as No. 9.

Citric acid gives but little promise as a solvent for available potash. The virgin soil of average fertility contains less potash soluble in citric acid than soil No. 1, which is deficient in available potash.

Sufficient results were not secured to determine the value of the N/5 $CaCl_2$ method. This method, however, possesses one serious drawback—the presence of a large amount of lime in the solution, the last traces of which are difficult to remove, requiring excessive washing to separate the potash. The relatively large amount of lime and small amount of potash in the solution make the determination of the latter quite difficult.

LIME.

The use of carbonated water as a solvent for lime, as proposed by Hallemann (Landw. Vers. Stat. 41), was tested in the Minnesota Experiment Station laboratory, with the following results:

	Soil No. 7. Deficient in lime.	Soil No. 8. Virgin soil, average fertility.	Soil No. 9. Virgin soil, high fertility.
	Per cent.	*Per cent.*	*Per cent.*
CaO	0.018	.....	.....
	.019	.....	.....
	.022	0.06	0.80

Fifty grams of soil No. 1 and 100 mg of $CaCo_3$ gave 0.079 per cent CaO, the theoretical per cent, making no allowance for fixation changes, being 0.125. As no other results were received, no conclusion can be drawn regarding the value of this method.

NITROGEN.

[Total nitrogen by official method.]

Analyst.	Soil No. 3.—Deficient in available nitrogen.		Soil No. 4.—Deficient in available nitrogen.		Soil No. 8.—Virgin soil, average fertility.		Soil No. 9.—Virgin soil, high fertility.	
	Total nitrogen.	Humus nitrogen.	Total nitrogen.	Humus nitrogen.	Total nitrogen.	Humus nitrogen.	Total nitrogen.	Humus nitrogen.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
Frank Shutt, Canada	.....	.....	0.049	0.029	0.253	0.156	0.581	0.29
Harry Snyder, Minnesota	0.03	0.01	.046	.017	.25	.168	.59	.25

The use of 3 per cent NaOH as a solvent for the humus nitrogen, after treating the soil with dilute HCl and distilled water, gave concordant and closely agreeing results.

SEPARATION OF COARSE AND MEDIUM SAND.

When the soil samples were distributed it was the plan to procure sieves with circular holes 0.5 and 0.25 mm in diameter for the separation of the coarse and medium sand. To insure accuracy, it was the desire to have the different sets of sieves bored with the same drill, so that the results would be more comparable. No dealer in chemical supplies was found who would agree to make the sieves for less than $100 per set. Sieves made from perforated metal were finally obtained, but they were sent to the analysts too late for any results to be secured. The separation of the various grades of soil particles is a matter which has not been taken up by this association. There are many problems connected with the mechanical analysis of soils which should receive careful consideration.

The diameters of the different grades of particles should be established on a proper basis and not on a conventional one. That is, there should be a constant ratio between the diameters of the different grades of particles. Mr. Hopkins, of the Illinois Experiment Station, proposes as a constant ratio the square root of 10. The adoption of such a scale would enable us to properly define the various grades of the soil particles. The diameters of the different particles should vary according to a constant ratio. At present the ratios of the diameters vary from 2 to 5, which does not place them on a comparable basis.

The adoption of a constant scale for the diameters of the different particles is as important in soil work as the metric system is for chemical work. In the mechanical analysis of soils the results should be given on the basis of the dry soil, as in the chemical analyses, and not on the basis of the air-dry soil.

There are other problems in the mechanical analyses of soils which should also receive attention, but for the present I consider that the two matters mentioned are the most important.

RECOMMENDATIONS.

The following recommendations are offered:

I. That the use of a fifth normal solution of hydrochloric acid, as proposed by Goss, be adopted as a provisional method for the determination of the more active forms of the phosphoric acid in soils.

II. That for the humus nitrogen determination a 3 per cent solution of NaOH be used as a solvent for extraction after treating the soil with 2 per cent HCl and washing with distilled H_2O. This simply defines the strength of the solution and leaves the analyst free to apply the principles in any way that he may desire. It is desirable to have the strength of the solutions defined, so that results will be more comparable. Experiments have shown that more constant results are secured with a 3 per cent solution than with one of any other strength.

III. That the use of $CaCl_2$ and alkaline NH_4Cl as solvents for potash be discontinued, and that for the coming year the so-called International European method for the determination of available potash be given a trial.

IV. That Hallemann's method for the determination of the active lime compounds of the soil be given a further trial during the coming year.

The referees desire to thank the various analysts who have taken a part in the work.

The recommendations of the referee were referred to the committee. The supplemental report on ash analysis was called for, and read by Mr. Hartwell.

SUPPLEMENTAL REPORT ON ASH.

By Burt L. Hartwell, *Associate Referee.*

At the request of the referee on soil and ash analysis I have relieved him of the duties connected with the preparation and mailing of ash samples and compiling and discussing the results obtained from the chemists who have kindly cooperated in the work.

The hay used in obtaining the ashes was taken from the mow, care being exercised that no more dirt be admixed with the ash than may have adhered to the hay in the natural course of curing. It is not probable, under the circumstances, that the sample of ash contained more than 1 per cent of sand. The average amounts of insoluble silica, as determined by the association on the samples of ash distributed during 1895, 1896, and 1897, have been 41.56, 5.98, and 12.05 per cents, respectively. In view of the conflicting results which have been obtained in the separation of soluble and insoluble silica, it becomes very desirable to know the probable amount of foreign material which is contained in the sample distributed, as a guide in questioning reported results. As a large amount of insoluble matter would be out of place in plant ashes intended for scientific investigation, it would seem advisable that care be taken to exclude the same from samples intended for cooperative work.

In the preparation of the ash no precaution was taken by burning at a low temperature to prevent the volatilization of potash and soda. The conditions under which the ignition was carried on were such that the manganese was converted into the form of manganic acid, as shown by the green coloration imparted to the ash and aqueous solution, due to potassium and sodium manganate, and the pink color exhibited, upon the subsequent addition of acid, by potassium permanganate thus formed. The manganese has therefore been recorded in the following analyses as MnO_2, instead of the more usual form of Mn_3O_4.

The following circular letter was sent, with samples of ash, to Elton Fulmer, Pullman, Wash.; F. W. Traphagen, Bozeman, Mont.; Harry Snyder, St. Anthony Park,

Minn., and the Division of Chemistry, Department of Agriculture, Washington, D. C., who had expressed a wish for the same:

KINGSTON, R. I., *May 25, 1898.*

DEAR SIR: I send you to-day by mail a sample of ash prepared from a mixed hay, consisting principally of orchard grass (*Dactylis glomerata*). The ash was bottled hot, and a moisture determination should be unnecessary. A complete analysis is desired, following the methods described in Bulletin 46, page 43, Division of Chemistry, United States Department of Agriculture.

Considerable difficulty having been experienced by the association in separating the soluble from the insoluble silica, and as the process is important on account of being essential to the determination of pure ash, a sample has been selected which is rich in soluble silica and will require every precaution for its correct determination. Anyone wishing a more detailed discussion of the method will do well to consult Fresenius's Quantitative Chemical Analysis, pages 420–421, second American edition, where several important precautions are mentioned. Our methods do not specify the amount of water which may be used in connection with the saturated sodium carbonate solution. Two volumes of water to each volume of the saturated solution are recommended. Please state whether the direct or indirect method was used in determining the soluble silica. Caution is necessary, where the amount of silica in the soluble form is as large as in the sample sent, to prevent its changing to the insoluble form at an undesired time.

As no method is given for the determination of iron and alumina in ashes, the following method from Crooke's Select Methods in Chemical Analysis, page 539, second edition, is recommended for trial: The filtrate from the precipitate of ammonium phosphomolybdate obtained during the determination of phosphoric acid is "cautiously neutralized with ammonia, care being taken that the temperature does not rise above 40° C. and that the alkali is added only in slight excess; allow to stand in a warm place until the precipitate completely settles, filter the clear supernatent fluid, wash by decantation, then transfer it to a filter, and finish the washing. The precipitate is dried, ignited, and weighed as ferric oxid and alumina."

Previous to weighing the potassium and sodium chlorids evaporation must be repeated until the residue is completely soluble in water. It is advisable to add a few drops of hydrochloric acid before the final evaporation.

Please report results as soon as obtained, describing any deviations which may have been made from the prescribed methods. Suggestions and criticisms concerning the methods are especially desired.

Yours, very truly,

BURT L. HARTWELL,
Associate Referee.

Ash sample.

Element.	J. K. Haywood, Washington, D. C.	B. L. Hartwell, Rhode Island.	Mr. Hummel, Minnesota.[1]
	Per cent.	*Per cent.*	*Per cent.*
Soluble SiO_2	15.87	15.76	4.18
Insoluble SiO_2	.89	.89	11.88
Total SiO_2	16.76	16.65	16.06
K_2O	36.21	35.42	.....
Na_2O	3.55	3.70	7.62
CaO	11.97	11.62	.....
MgO	5.78	5.74	.....
MnO_3	1.03	.96	.....
Al_2O_3	.58	.95	.....
Fe_2O_3	.30		.....
P_2O_5	9.83	9.90	10.80
SO_3	3.55	3.72	3.64
Cl	12.70	12.56	12.87
C	.14	.06	.89
CO_2	1.03	1.24	1.58
Total	103.23	102.52	.....
O equivalent to Cl	2.86	2.84	.....
Corrected total	100.37	99.68	.....

[1] These results were obtained by an advanced student, inexperienced in ash analysis, and were not intended to serve as a test of the method.

Mr. Haywood remarks that the "sand and silica were determined together. The sand was then determined with the carbon and subtracted from the combined sand and silicia.

"I find it exceedingly difficult to get rid of the foreign matter in determining the chlorids of sodium and potassium."

Mr. Hummel used the direct method in the determination of soluble silica. He reports that "the K_2O results were unsatisfactory."

I determined the soluble silica by the direct method of evaporating the alkaline filtrate to dryness. Repeated precipitation and evaporation were necessary to obtain the alkaline chlorids in a pure state entirely soluble in water.

As a committee was appointed at the last meeting of this association to carefully rewrite the methods of ash analysis, suggestions from me as to changes in the same would be inappropriate at this time.

In the report of the referee on soil and ash analysis for 1897 the determination of soluble silica was mentioned as being of very doubtful utility, and a recommendation was offered to omit the same. The separation, nevertheless, of soluble from insoluble silica is necessary in obtaining the amount of pure ash in a given sample, i. e., the amount of ash ingredients free from carbon, carbon dioxid, and sand (insoluble silica). An analysis of so-called ash which furnished no indication of the amount of foreign material present would be of doubtful scientific value. The procedure as indicated in the methods of the analysis of the association is, I believe, capable of yielding more uniform results than have been heretofore reported. It may be necessary to introduce into the method such minute directions and precautions as are absolutely essential to correct results before the inexperienced will be able to make a determination which will be looked upon as a test of the method rather than of the chemist.

The method given in the above letter for the determination of ferric and aluminic oxids seems very satisfactory and worthy of further trial.

Opinions agree as to the extreme tediousness in obtaining the chlorids of potash and soda free from impurities in many samples of ashes, and it is hoped that your committee will recommend some changes in the method in this respect.

As considerable difficulty exists regarding the determinations of silica and the alkalies, and as concordant results, if secured, would not necessarily represent the correct percentages of these ingredients, it would seem to be desirable to adopt the idea followed by recent referees on potash and prepare an artificial ash containing known amounts of those consituents the determinations of which are especially worthy of study.

[The methods for the analysis of ashes transmitted by the referee will be found in the revised edition of Bulletin No. 46, Division of Chemistry.]

At the conclusion of the report the president called for papers on this subject, and the following were submitted:

PROBLEMS IN SOIL INVESTIGATIONS.

By Harry Snyder.

Some objections have been raised against the use of HCl, 1.115 sp. gr., as a solvent in soil analysis because it fails to give the available plant food. It is argued that one soil may contain 0.40 per cent potash soluble in HCl, 1.115 sp. gr., and be deficient in available potash, while another soil may contain 0.20 per cent and be liberally supplied. While this is frequently true, it does not follow that HCl has no value as a solvent in soil analysis.

While dilute organic acids and other solvents are valuable to determine the degree of solubility of the materials in the soil, they fail to give any insight into the chemical character of the soil. With the results from both HCl and dilute acids before him, the chemist is able to form a better idea of the true nature of the soil than if

either results were considered alone. With HCl as a solvent, if 0.20 per cent CaO and 0.15 per cent Co_2 be obtained, there is no necessity for making an available lime determination with carbonated water. If 0.20 per cent CaO be obtained and only a few hundredths of a per cent CO_2, then there is reason to suspect poverty in available lime.

The hydrochloric acid solvent is valuable in showing an unbalanced condition of the plant food in a soil. For example, let us compare two soils.

	Soil A.	Soil B.
	Per cent.	*Per cent.*
Total insoluble matter	82.40	80.25
K_2O	.40	.25
CaO	.25	.25
P_2O_5	.18	.15
CO_2	.02	.16
SO_3	.01	.03
Total nitrogen	.15	.12
Humus	2.60	1.88

Field tests have shown that soil A is deficient in available K_2O, and that there is no large amount of either available N or P_2O_5. How are the field and laboratory results to be reconciled? Soil A, with 0.4 per cent K_2O, is in need of available K_2O, while B, with 0.25 per cent, is liberally supplied.

In soil A the low per cent of CO_2 and of SO_3 shows that the lime is not present as carbonate or sulphate, while in B it is present mainly as carbonate. In soil B there is a more balanced condition of the elements. The presence of the lime carbonate enables the potash to be utilized, because it furnishes basic material to take the place in the soil of the potash removed in the crop. The conditions are more favorable for active nitrification in B, and the organic matter can not produce acid products. The presence of the neutral organic matter in this soil exerts a favorable influence upon the availability of the phosphoric acid. In soil B there are less of the acid soluble plant food elements than in A, but there is a better balanced condition of the plant food, which renders it more available. The fact that a standard can be established as to the minimum amount of acid soluble plant food a soil should contain does not materially detract from the value of the method. An isolated soil analysis, like an isolated analysis of a well water, may fail to give all of the information desired. When, however, systematic analyses are made of the well waters in any region, valuable results are secured. So with soil analysis, the most valuable results are secured when a number of fertile soils in a locality are examined, and then unknown soils from the same locality are compared with those of known fertility.

Many stations have hesitated to undertake soil investigations, fearing that the results secured would not compensate for the efforts made. If one-half of the energy which is now spent in the analysis of fertilizers were directed to soil investigations, there is no question but that valuable results would be obtained.

In working out methods to determine the degree of solubility of the various plant food elements the methods should apply to all kinds of soils. A significant letter from Dr. Hilgard touches upon this point. He states that in the selection of soils for the association work, virgin soils should be represented, particularly that class composed mainly of silt particles and containing only a small amount of clay particles. In such soils crops feed more liberally upon the mineral matter combined with organic compounds, and ordinary methods for determining available plant food are not applicable to such soils. Grandeau's methods for the analysis of such soils is the most satisfactory one that has yet been proposed.

The joint use of dilute acids to determine the active forms of phosphoric acid and potash; of carbonated water to determine available lime, and, of HCl, 1.115 sp. gr., to give an insight into the chemical nature of the soil, give results which enable the chemist to determine with a fair degree of accuracy the condition of the soil as to available plant food. As to available nitrogen, it would seem best to approach this problem in a different way. The soil should first contain a fair amount of total nitrogen; and, second, the soil should be normal in composition, so that the nitrogen may be rendered available. The total nitrogen, the humus nitrogen, and the lime soluble in carbonated water, when determined and the results are properly interpreted, give valuable indications as to the availability of the soil nitrogen.

The term "active forms of plant food" should be applied to the phosphoric acid and potash soluble in dilute acids, in preference to the term "available plant food," which is so frequently misused in commercial-fertilizer literature.

The acid or basic characteristics of soils are also important matters to consider. In some soils there are excessive amounts of alkaline compounds, while in others there is not a sufficient amount to prevent the formation of organic acids. The methods of soil investigations should be suited to the study of both classes of soils. Methods which are applicable to one class of soil sometimes fail when applied to other soils. For example, soil No. 9 contains 3.15 per cent CaO_2 and 4.19 per cent CO_2, which make theoretically over 7 per cent $CaCO_3$. When this soil is extracted with NH_4OH for the determination of the free humic acid a dark brown solution is obtained, which, when neutralized with an acid, gives from 0.3 to 0.4 per cent of weighable free humic acid products. When the soil is extracted with carbonated water, 0.8 per cent CaO is obtained. The soil is liberally supplied with lime carbonate, gives an indifferent reaction, and yields 0.3 to 0.4 per cent of so-called free humic acid and produces exceedingly good crops. Evidently the method for the determination of free humic acid is not applicable to this soil.

Another important question in soil investigations is, What standard shall we take for determining the deficiency of any element? Many have answered the question by saying that field tests should be made the standard. But are simple field tests always reliable? To illustrate: Acid phosphate is used and good results are secured; the conclusion is drawn that the soil is deficient in available P_2O_5. The fact that calcium sulphate is an invariable constituent of the phosphate is not considered. The action of the sulphate upon the soil potash and nitrogen may have been equally as important a factor in the crop production as the available phosphate; in fact, field tests are not infallible.

In rewriting the directions for soil analysis the committee appointed at the last meeting of this association should be empowered to simplify the present official method. There is no necessity for first precipitating the lime, then dissolving it and reprecipitating it. One precipitation with careful washing is sufficient. The volumetric method for the determination of CaO should be made an alternate method. The method is a rapid and reliable one; in fact, the ignition of a calcium oxalate precipitate is not an easy matter, as some lime may be reduced to an oxid while part is in the form of a carbonate. There is no special reason why the determination of manganese should be made, and there is nothing gained by making a separation of the iron and aluminia. By making our present method more direct and omitting the unnecessary determinations, soil analysis will be relieved of some of its undesirable features.

DANGER OF DRAWING ERRONEOUS CONCLUSIONS FROM PLANT SOIL TESTS.

By H. J. Wheeler.

The data to be presented here were obtained in connection with a soil test, by means of plants and fertilizers, which has been continued without interruption for nine consecutive years at the Rhode Island Agricultural Experiment Station. The test has been conducted upon eight twentieth-acre plots, separated from one another by paths 3 feet wide. It comprised a plot receiving no manure, one treated with nitro-

gen, one with phosphoric acid, one with potash, one with nitrogen and phosphoric acid, one with nitrogen and potash, one with potash and phosphoric acid, and one with all three manurial substances. Owing to some inequalities on one portion of the unmanured plot, and to certain irregularities in the treatment of that which received all three manures, their detailed consideration here has been omitted, since it appears unnecessary to the purpose of this paper. It should be stated, however, that almost without exception the best crops have been obtained by the combined use of all three substances, and the poorest ones where no manure was applied, though in certain seasons but little advantage has been gained from the use of either nitrogen or potash when applied singly.

To those plots where the manurial substances were applied either separately or in connection with others the rates per acre for the various years have been as indicated below:

Material.	In 1890 and 1891.	In 1892 and subsequently.
	Pounds.	*Pounds.*
Nitrate of soda	150	480
Dissolved boneblack	350	600
Muriate of potash	150	200

From 1890 to 1893, inclusive, the entire plots were devoted exclusively to maize. In 1894 thirty-seven varieties of plants were employed, two rows of each variety having been run crosswise of the plots, with the hope of obtaining an idea as to whether they would all give similar indications as to the needs of the soil. In pursuance of the same idea, a few of the varieties which gave the most dissimilar results in 1894 were again tested in 1895, 1896, and 1897. In 1895 maize was not included among the plants tested, which explains the absence of results for that year in the succeeding table. On account of the large number of plants employed, the area devoted to maize in 1894 was small, though it was largely increased in 1896 and 1897. Owing to the remarkable character of the results in 1897 the entire plots were once more devoted exclusively to maize in 1898, in order to procure the fullest data possible and to furnish a better comparison with those obtained in the earlier years before other crops were introduced into the experiment.

The following is a tabulation of the results secured with maize during the eight years when that crop was grown:

Table showing the relative weights (in pounds) of maize obtained in a soil test in the years before and after liming the soil.

[In the year 1895 maize was not grown upon the plots.]

Plot No.	Manures.	Yields before liming (corn and stover).					Yields after liming (corn and stover).		
		1890.	1891.	1892.	1893.	1894.[1]	1896.[2]	1897.[3]	1898.
1	Nitrogen	180.0	60.0	60.0	50.0	26.0	10.6	103.3	215.0
2	Phosphoric acid	210.0	129.0	130.0	145.0	88.0	20.8	32.6	130.0
3	Potash	293.8	107.5	61.0	112.5	28.0	11.9	133.8	295.0
4	Nitrogen and phosphoric acid	273.8	139.5	205.0	122.5	116.0	38.5	180.3	350.0
5	Nitrogen and potash	282.5	112.5	88.0	102.5	32.0	26.8	222.5	395.0
6	Potash and phosphoric acid	293.8	157.8	230.0	163.8	202.0	49.2	230.8	335.0

[1] Yield from two rows only, running crosswise of the plots.

[2] Limed in the spring of 1896 and planted immediately. Yield from eight rows only, running crosswise of the plots.

[3] Yield from eight rows only, running crosswise of the plots.

In 1890, as may be seen from the foregoing table, a considerable amount of readily assimilable phosphoric acid must have been at the disposal of the plants, since potash appeared to be the most deficient manurial substance.

From 1891 to 1894, inclusive, the plots receiving single manures, as well as those to which combinations of two were applied, showed without exception that phosphoric acid was by far the most deficient ingredient.

It will be noticed that the results in 1896 (the season in which air-slacked lime was applied to all of the plots at the rate of 2½ tons per acre) point, as in the foregoing years, to a greater deficiency of phosphoric acid than of nitrogen or potash. The two succeeding years, however, the plots receiving single manurial substances only, and those where combinations of two manures were employed, gave every indication that the plants were no longer in great need of phosphoric acid. In fact, in 1898 the yield from the potash and nitrogen plot, where maize had sometimes made but little growth, was the greatest obtained from any of the plots.

Certain cereals, which, like maize, had shown in every particular in 1894 that the soil was most deficient in phosphoric acid, did practically as well in 1895 upon the plot receiving potash and nitrogen as upon that treated with nitrogen and phosphoric acid. However, on account of the fact that they had agreed so thoroughly with maize in 1894, and for the reason that in 1895 the growth upon the nitrogen as well as upon the potash plot was so much inferior to that where only phosphoric acid was used, it was concluded that by mistake potash and nitrogen must have been applied in 1895 to the nitrogen and phosphoric acid plot (No. 4), while the manures intended for that plot were probably applied to plot 5, which should have received nitrogen and potash. Nothing has since transpired to change this view. Even if such an interchange resulted, this factor can not be used as an argument against the conclusion that lime aided by a large amount of moisture was probably the prime agent in producing the remarkable results of the last two years. This is evident when one observes that the yield in 1896 upon the plot receiving nitrogen and potash had again fallen far behind that on the adjoining one which received nitrogen and phosphoric acid. Even if, in spite of this evidence, one were still inclined to attribute the changed character of the results on these two plots in 1897 and 1898 to an after effect due to a possible interchange in the manures in 1895, this would not furnish an explanation of the remarkable growth of the maize in the last two years upon the plots where nitrogen and potash were applied singly.

Taking into consideration, therefore, every objection to such an idea,[1] one is forced to the conclusion that the lime had probably in some way liberated and placed at the disposal of the crop large quantities of phosphoric acid which were formerly almost useless and not in condition to be assimilated by plants. It must be evident from what has been shown that if one has a soil which, for either physical or chemical reasons, needs lime, correct conclusions as to its probable manurial requirement after liming are not likely to be obtained by a soil test from which lime is omitted, and which is continued for but one or two seasons.

Since it would be irrational to attempt to cultivate land sorely in need of lime without resorting to its use, the recognition of the existence or absence of soil acidity as an indication of whether calcium carbonate is deficient or not seems to be a step which should in certain soils precede the inauguration of a soil test.

In virgin soils Hilgard finds that much over 0.1 of 1 per cent of phosphoric acid, as determined from solutions obtained by digesting the soil for five days with hydrochloric acid of 1.115 specific gravity, is enough to insure the profitable production of crops for many years without resort to phosphatic manures. By digestion with acid

[1] It is possible that the gypsum in the superphosphate may have exerted a favorable influence upon the growth of the maize, but when one considers that the soil contained more than 0.5 of a per cent of lime (CaO) in various forms, and that in other experiments on adjoining plots the use of air-slacked lime was of no particular advantage to the crop, there seems to be little probability that the wonderful effect from the use of superphosphate before liming could have been attributed chiefly or entirely to the gypsum instead of to the phosphoric acid.

of the above strength for ten hours, the average of several determinations by members of the Association of Official Agricultural Chemists in the soil of plots 2 and 5 in this test, showed 0.202 and 0.191 per cent, respectively, or about double the amount which Hilgard finds ample for many years in virgin soils. It is apparent, therefore, that the fault of this soil lies not in a small percentage of phosphoric acid, but in the form in which it exists in the unlimed acid soil.

In 1895 this soil was selected by the reporter on soils and ash for the Association of Official Agricultural Chemists on account of its evident deficiency in phosphoric acid, for use in comparative tests for the purpose of obtaining a reliable chemical method by which the manurial requirements of soils long under cultivation could be ascertained. So far as tests with plants had till then shown, a better soil could hardly have been obtained for the purpose, yet after liming it has been found to be but slightly in need of phosphates. This seems to emphasize the fact that a soil test involving the use of potash, phosphoric acid, and nitrogen only may not always prove an accurate guide as to the manurial treatment, nor in the selection of soils designed to serve as a basis for judging of the relative merits of various methods of chemical soil analyses. It appears, therefore, that each plot should probably receive such an amount of lime or other compounds as may, for chemical and physical reasons, be required to put the soil in the best condition in other respects before the test as to relative deficiency of nitrogen, potash, and phosphoric acid is made.

In view of the presence, in many of the States of the Union, of soils lacking calcium carbonate, these factors appear to be worthy of general recognition, since their influence upon the practical and scientific deductions based upon soil tests may in many cases be of great importance.

ESTIMATION OF THE LIME, POTASH, AND PHOSPHORIC ACID IN HAWAIIAN SOILS PROBABLY AVAILABLE FOR THE IMMEDIATE CROP.

By Walter Maxwell, *Hawaiian Experiment Station.*

ASPARTIC ACID METHOD.

(1) The hypothetic principle of this method rests upon the assumption that the solvents used shall compare with the solvents that operate in the processes of soil disintegration and plant-food preparation in the field. (See the author's work on the Lavas and Soils of the Hawaiian Islands; also American Chemical Journal, Vol. XX, No. 2, p. 107.)

(2) The standards of control of this method are the ascertained results of the natural processes of soil decomposition in the field, under the conditions of climate and nature of the soils of the Hawaiian Islands, as indicated by the "results of cropping" and "the composition of the waters of discharge" flowing from the land into the sea. (See Lavas and Soils of the Hawaiian Islands.)

(3) The sample is obtained by the mode given in previous publications, viz, by the pipe where this is practicable, otherwise by the spade. The calculation of available matter per acre is based upon the specific gravity (weight of the cubic foot) of the soil and the depth to which the sample is taken. In thin soils the amount is less, in deep soils greater, than the amount found in 1 acre to a depth of 1 foot, upon which depth calculations so far in the use of this method have been based.

(4) The solvent used in this method is aspartic acid. This acid was adopted because it was found that "aspartic acid dissolves phosphoric acid, lime, potash, and other bases (iron and alumina) out of the soil in almost the exact proportions that these elements have been found in the 'waters of discharge,' and in which they are being removed by 'cropping.'". (See Lavas and Soils of the Hawaiian Islands, pp. 179–181.)

(5) The method established and used so far, but which the author may modify in some features, is as follows: Two hundred grams of whole soil (not fine earth), air dried, was put into a glass-stoppered Winchester quart bottle; to this soil were added 1,000 cc of distilled water containing 10 grams of pure aspartic acid, thus making a 1 per cent solution. The bottle was gently shaken every fifteen minutes during the day

portion of twenty-four hours (from 8 o'clock a. m. to 5 o'clock p. m.). By "gently shaken" is meant that the bottle was taken up and shaken in circular movement until the soil at the bottom was totally and thoroughly moved, but without ever rising above the surface of the solution, thus avoiding a remnant of the soil attaching to the sides of the bottle above the solution surface. This explanation is given because it has been shown that in "shaking the bottle containing the soil and solvent, which is the only mode of securing a uniform distribution and action of the solvent, the results of the action will be in large measure proportionate to the mode and vigor of the shaking." (See Lavas and Soils, pp. 136–138.) At the end of twenty-four hours the contents of the bottle were emptied upon a filter, when 750 cc of the clear filtrate was taken for analysis. The 750 cc of filtrate was evaporated to dryness and the residue gently ignited to remove all organic matter. The mineral residue was moistened with hydrochloric acid, evaporated, and dried, then again softened with the same acid, warmed, and taken up with water and filtered. After removal of iron and alumina from the filtrate with ammonia, the lime and potash were estimated in the usual way. The ammonia precipitate, containing also the iron and alumina, was dissolved in a little hydrochloric acid, neutralized with ammonia, the solution cleared with a little nitric acid, after which the phosphoric acid was precipitated and estimated by the common practice.

(6) The estimation of the amounts of the elements that are "probably available for the immediate crop" is drawn from the foregoing analysis: As "a 1 per cent solution of aspartic acid takes out of Hawaiian soils in twenty-four hours the same amounts of lime, potash, and phosphoric acid that are removed during the production of ten crops of cane, therefore one-tenth of these amounts may be taken as a nearest approach to the proportions of lime, potash, and phosphoric acid that are available for the immediate crop." (See Lavas and Soils, p. 181.)

As it has been said, the principle of this method rests upon the assumption that the solvent used shall compare with the solvents that are actually operating in nature; and that the standards of control of the results of this method are the ascertained results of the natural processes of soil—decomposition in the field. Not any consideration has made it appear advisable to the author that a solvent should be used which is not found amongst the acids that result from vegetable existence and decay, and which consequently is not used by nature in the field. It has, on the other hand, appeared to him absolutely necessary that, before concluding that the results obtained by the use of any artificial method of examination are rational and of any authority and value, these results should be subjected, if possible, to comparison with the standard of results actually following from the processes operating in the field.

A PLEA FOR A SCIENTIFIC BASIS FOR THE DIVISION OF SOIL PARTICLES IN MECHANICAL ANALYSIS.

By C. G. Hopkins.

The following is the system of divisions which appears to be rapidly coming into use in mechanical soil analysis:

Division No.	Size of particles.
	mm in diameter.
1	> 1
2	1 to .5
3	.5 to .25
4	.25 to .1
5	.1 to .05
6	.05 to .01
7	.01 to .005
8	< .005

These limits were originally chosen by mere chance and have no more of a scientific basis than have the common systems of weights and measures or the Fahrenheit thermometer scale. They were first employed by Dr. Thomas B. Osborne in a study of methods of mechanical soil analysis and were not intended by him to be used in a practical study of soils. Regarding this, Dr. Osborne says:

"In working out the beaker method of soil analysis I employed the limits of the various grades with reference simply to convenience in using my eyepiece micrometer. I have always thought that the limits of the various grades should be determined by a careful consideration of the various conditions involved in the problem of proper mechanical analysis of a soil, and have been surprised to see that the arbitrarily chosen limits of the various grades employed by me have been followed by others in applying the method in practice."

The one very serious objection to this system is that the ratio of the diameter of the largest particles to that of the smallest particles in each of the various divisions is not a constant. In divisions 2, 3, 5, and 7 the ratio of largest to smallest diameter is 2 to 1; in division 4 the ratio is 2.5 to 1, while in 6 the ratio is 5 to 1. As a consequence of this wider ratio, division 6 will receive twice as much soil as division 4 and two and one-half times as much as either 2, 3, 5, or 7 from a sample whose particles are in uniform gradation. In practical work, as a rule, a much larger percentage of soil is found to fall in division 6 than in either 5 or 7, and this is plainly not due to any peculiarity of the soil, but to the simple fact that by definition the limits for division 6 are two and one-half times wider than those of either contiguous division.

The following table shows the ratios of diameters, of surfaces, and of volumes of the limiting soil particles in each division except those at the ends of the series. It also gives the theoretical composition of a sample which is made up of soil particles of uniform gradation between the limits of gravel and clay, assuming the end divisions to contain the average percentage.

Division No.	Size of particles (mm.).	Ratio of diameters.	Ratio of surfaces.	Ratio of volumes.	Theoretical percentage composition.
1	> 1				12.50
2	1 to .5	2 to 1	4 to 1	8 to 1	9.68
3	.5 to .25	2 to 1	4 to 1	8 to 1	9.68
4	.25 to .1	2.5 to 1	6.25 to 1	15.6 to 1	12.10
5	.1 to .05	2 to 1	4 to 1	8 to 1	9.68
6	.05 to .01	5 to 1	25 to 1	125 to 1	24.20
7	.01 to .005	2 to 1	4 to 1	8 to 1	9.68
8	< .005				12.50
Total					100

The differences in the ratios of the largest to the smallest particles in the several divisions become even larger when we consider surfaces or volumes instead of diameters, and yet such phenomena of soil physics as capillarity and porosity are in relation to the surfaces or volumes of soil particles rather than to their diameters. It will also be observed that the percentages of soil in the different divisions vary from 9.68 to 24.20, although by theory the soil is of uniform gradation.

Because of this objection to the common system of making the divisions in mechanical soil analysis, the system illustrated in the following table was worked out by the writer some two years ago. It was subsequently adopted by the department of soil physics of the University of Illinois, and has since been employed with entire satisfaction.

The table shows the theoretical composition of a soil whose particles are in uniform gradation within the limits of the system, assuming the end divisions to

contain the average percentage. Ratios of largest to smallest particles are also given.

Division No.	Name of particles used.	Size of particles (millimeters).	Ratio of diameters.	Ratio of surfaces.	Ratio of volumes.	Theoretical percentage composition.
1	Gravel	> 1				12.50
2	Coarse sand	1 to .32	3.2 to 1	10 to 1	32 to 1	12.50
3	Medium sand	.32 to .1	3.2 to 1	10 to 1	32 to 1	12.50
4	Fine sand	.1 to .032	3.2 to 1	10 to 1	32 to 1	12.50
5	Coarse silt	.032 to .01	3.2 to 1	10 to 1	32 to 1	12.50
6	Medium silt	.01 to .0032	3.2 to 1	10 to 1	32 to 1	12.50
7	Fine silt	.0032 to .001	3.2 to 1	10 to 1	32 to 1	12.50
8	Clay	< .001				12.50
	Total					100

The square root of 10 (approximately 3.2) is adopted as a common factor in passing from smallest to largest particles in all divisions of defined limits, and thus the ratio of the largest to the smallest particles becomes a constant; this applies to the ratios of diameters, of surfaces, and of volumes. Every division receives the same percentage of a soil of uniform gradation. This system employs 0.001 mm (diameter) as the limit of separation between fine silt and clay. (By the use of centrifugal force, as described in another paper, this separation is rendered entirely practicable.) Three grades of silt are thus determined instead of two as by the ordinary system, while the four grades of sand usually determined are here condensed to three, although the limit of separation between sand and silt is practically the same in either system. Both systems employ as limits of divisions 1, 0.1, and 0.01 mm (diameter), and it is important to note that, by combining the percentages obtained in practical work so as to make four divisions instead of eight, the two systems become identical and of course strictly comparable to that extent.

With the proper accessories the microscopic measurements are made with the same degree of convenience and exactness in either system.

The important fact should be mentioned that the new system readily admits of expansion. By taking the fourth root of 10 (approximately 1.8) instead of the square root of 10, each of the limited divisions is simply divided into two equal divisions. This will make a series of fourteen separate divisions of soil particles and the system still retains all of the advantages which have been mentioned. The following form is recommended for use with the expanded system:

Division No.	Name of particles.	Size of particles (millimeters).
1	Gravel	> 1
2	Coarse sand	1 to .56
		.56 to .32
3	Medium sand	.32 to .18
		.18 to .1
4	Fine sand	.1 to .056
		.056 to .032
5	Coarse silt	.032 to .018
		.018 to .01
6	Medium silt	.01 to .0056
		.0056 to .0032
7	Fine silt	.0032 to .0018
		.0018 to .001
8	Clay	< .001

A RAPID METHOD OF MECHANICAL SOIL ANALYSIS, INCLUDING THE USE OF CENTRIFUGAL FORCE.

By C. G. Hopkins.

A sample of 10 grams of soil is placed in a small sieve having circular perforations 0.32 mm in diameter. (Such a sieve is easily made by carefully enlarging the perforations of a 0.25 mm sieve with a drill of 0.32 mm diameter.) The sieve is placed in a porcelain evaporator and the fine earth washed through with successive portions of water. The sieve should reach nearly to the bottom of the evaporator. The gravel and coarse sand remain in the sieve (under water) and are rubbed with a rubber pestle till clean from the fine earth, which is gradually transferred with the successive portions of water from the evaporator to a bottle of 500 cc to 600 cc capacity.

The gravel and coarse sand are dried, separated by a millimeter sieve, ignited, and weighed.

The bottle containing the fine earth and 300 cc to 400 cc of water is placed in a shaking machine similar to that described by Professor Whitney in Bulletin No. 4, page 9, Division of Soils, United States Department of Agriculture. The bottle is shaken for a length of time deemed to be sufficient for complete disintegration of the soil. About 125 cc of the contents of the bottle are then transferred to a flat-bottom glass cylinder about 40 mm in diameter and 160 mm high. The cylinder is placed in the centrifugal machine and whirled until all particles coarser than 0.001 mm diameter have been thrown to the bottom. The length of time necessary to accomplish this depends upon the diameter and speed of the machine. It must be determined by experiment for each machine with microscopic measurement of the particles remaining in suspension. With a machine of about 60 cm diameter and a speed of about 3,000 revolutions per minute not more than five minutes are required for this process.

A rubber stopper carrying two glass tubes similar to those used in the ordinary wash bottle is placed in the mouth of the cylinder, the longer tube reaching nearly to the bottom. By blowing in the shorter tube the water and suspended clay are transferred to a large beaker or bottle almost completely without disturbing the sediment. The sediment is then well stirred up, or "pestled," by means of a stout glass rod covered at the end with a rubber cap. Another portion of the contents of the shaking bottle is transferred to the cylinder, which is closed with a close-fitting solid rubber stopper and thoroughly shaken, the stopper being carefully rinsed off with fresh water. The cylinder is then whirled as before and the suspended clay removed. This is repeated until all of the fine earth has been transferred to the cylinder, fresh water being used as needed to complete the transfer. The sediment is again stirred up and shaken with fresh water, the cylinder whirled in the machine, and the suspended clay removed with the water, as before. This process is repeated until it is found that nothing remains in suspension with the speed and time adopted. The addition of a small amount of ammonia at the time the sediment is "pestled" is commonly recommended as hastening the process of separating the clay from the coarser particles. It has been used by the writer with apparent advantage.

The "clay water" will usually amount to three or four liters if the separation of clay is carried to a high degree of accuracy. It is evaporated in a large platinum or porcelain dish to a small bulk, the clay being then transferred with a small quantity of fresh water to a smaller dish, evaporated to dryness, ignited, and weighed.

The sediment remaining in the cylinder is shaken with fresh water and then whirled in the machine, preferably at a somewhat reduced speed, for a length of time sufficient to throw to the bottom all particles coarser than 0.0032 mm diameter, and the suspended fine silt removed with the water as already described. This process is repeated until nothing remains in suspension after whirling.

By further reducing the speed or time (or both) the medium silt, 0.01 to 0.0032 mm diameter, is also separated, after which gravity is substituted for centrifugal force, and first the coarse silt, 0.032 to 0.01 mm diameter, and then the fine sand, 0.1 to 0.032 mm diameter, removed, thus leaving in the cylinder only the medium sand, 0.32 to 0.1 mm diameter.

If the removal of clay has been complete, no "pestling" is necessary in making the subsequent separations.

The waters containing the medium and fine sand and the three divisions of silt are allowed to stand until the sediments have completely subsided. The clear water can then be removed by siphon almost completely. (It may be saved to be used again.) The various sediments are transferred to evaporators with small amounts of water, and after evaporating to dryness they are ignited and weighed.

Two analyses are carried on together and there is some advantage in carrying four samples at a time through the clay separation.

Moisture and organic matter are determined in another sample of 10 grams, and the mechanical analysis is then reduced to the basis of ignited soil, which is believed to be the only basis upon which such analyses are strictly comparable.

The following is a record of some of the analyses which the writer has made by this method and it is believed that it fairly represents its accuracy, although the error appears to be much less than is common in mechanical soil analysis:

Divisions.	Soil 1.	Soil 2.	Soil 3.	Soil 4.	Soil 5.	Soil 5.
Gravel	0.07	0.19	.....	.....	1.06	1.06
Coarse sand	1.54	1.64	0.44	1.03	6.36	5.83
Medium sand	6.91	7.66	1.52	3.78	29.01	28.71
Fine sand	7.28	7.64	3.98	8.38	9.43	9.64
Coarse silt	42.99	45.14	32.61	45.01	21.40	22.03
Medium silt	12.25	16.05	16.48	19.01	10.88	10.70
Fine silt	11.55	9.22	10.08	11.44	6.04	6.14
Clay	16.97	12.08	34.44	10.78	15.30	15.56
Total	99.56	99.62	99.55	99.43	99.48	99.67

The high degree of accuracy of this method is doubtless attributable to several conditions:

(1) A direct determination is made of the total clay.

(2) The total amount of water employed in a complete analysis is comparatively small.

(3) Analyses are easily completed on the next day after they are begun.

In all of these points the method differs essentially from the ordinary methods of mechanical soil analysis.

When it is understood that by the use of centrifugal force work may be done in five minutes' time which gravity will not accomplish in twenty-four hours, the value of this method is more fully appreciated.

It should be stated that, soon after this method had been worked out, the writer learned that Prof. Harry Snyder, of the University of Minnesota, was also using centrifugal force in mechanical soil analysis, although to a more limited extent than in the method herein described.

After the reading of the papers and a somewhat extended discussion, the report of the committee on recommendations was presented as follows:

SOILS AND ASH.

Your committee recommends the adoption of the first recommendation of the referee on soils and ash.

The committee recommends that the process described in the second recommendation of the referee be adopted as a provisional method.

The committee also recommends that the use of calcium chlorid as a solvent for potash be abandoned, that neutral or alkaline ammonium chlorid be given a further trial, and that for the coming year the so-called International European method for the determination of available potash be given a trial.

The committee also concurs in the fourth recommendation of the referee.

All the recommendations of the committee were unanimously approved.

The report of the referee on foods and feeding stuffs was then called for. The referee, Mr. W. H. Krug, read the following report:

REPORT ON CATTLE FEEDS.

By William H. Krug, *Referee.*

The work of Dr. Lindsey, the referee for the year 1897, had conclusively shown that all methods proposed for the estimation of starch, in which the material is directly subjected to the inverting action of acids, yield results which are above the actual starch content on account of the simultaneous conversion of the pentosans into pentoses and the galactan into galactose. He therefore recommended that the diastase method be made the provisional method for the determination of starch in all substances except in commercial starch and in potatoes. This recommendation was adopted, and the work on the determination of starch for this year was accordingly limited to the diastase method, as it seemed desirable, under the circumstances, to obtain as many results as possible by this method, whereby it would be given a severe test. It gives me pleasure to state that this has been to some extent realized, thirteen chemists reporting results obtained with the method.

The phenylhydrazin and phloroglucin methods for the estimation of the pentosans have again been compared and the method recommended for the estimation of galactan has been tried.

Three samples were sent out, No. 1 consisting of barley, No. 2 of bran, and No. 3 of red-clover seed. These materials were ground as fine as possible, thoroughly mixed, and immediately bottled and sealed.

In answer to the circular letter sent, 17 chemists agreed to cooperate with the referee. Twelve have returned reports.

I. The Estimation of Starch.

The provisional method, as adopted at the last session of the association, is described in the proceedings for the year 1897.

Samples 1 and 2 were chosen for the comparative work on this method, as the former consisted of a material rich in starch while the latter contained a relatively small amount of starch and a high percentage of pentosan.

The following table contains the results obtained on these samples:

TABLE 1.—*Results obtained with the diastase method.*

Analyst.	Barley.		Bran.	
	Air-dry sample.	Calculated to dry matter.	Air-dry sample.	Calculated to dry matter.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*
E. B. Holland, Mass. Exp. Station	66.58	73.70	8.39	9.11
C. D. Howard, N. H. Exp. Station	63.59	69.57	9.80	10.58
G. H. Jones, Vt. Exp. Station	68.49	75.62	[1]18.33	[1]19.79
G. L. Teller and J. F. Moore, Ark. Exp. Station	68.60	78.29	7.00	7.81
W. H. Heileman, Wash. Exp. Station	[1]57.06	[1]63.16	6.94	7.47
H. J. Patterson, Md. Exp. Station	[1]53.61	[1]59.81	[1]0.69	[1]0.74
F. S. Shiver, S. C. Exp. Station	66.17	72.92	8.56	9.24
H. C. Sherman, Columbia College, N. Y	63.84	71.33	11.01	12.01
O. C. Beck, Columbia College, N. Y	.....	.....	10.92	11.92
H. Fisher, Columbia College, N. Y	.....	.....	11.38	12.42
R. G. Wright, Columbia College, N. Y	64.72	71.64	.....	.....
S. S. Munson, Mich. Exp. Station	[1]53.73	[1]60.57	7.78	8.56
W. H. Krug, U. S. Dept. of Agr	65.45	72.76	7.88	8.61
Averages	65.93	73.23	8.97	9.77

[1] Excluded from the averages.

NOTES ON THE ESTIMATION OF STARCH.

E. B. Holland, Massachusetts.—The residues, when treated with iodin and examined microscopically, showed considerable starch in No. 1 and only a trace of starch in No. 2. The cuprous oxid was collected in sugar tubes and oxidized by heating in a continuous current of oxygen.

C. H. Jones, Vermont.—I found it necessary to use 90 cc of diastase solution to change all the starch.

G. L. Teller and J. F. Moore, Arkansas.—The work being so long delayed, freshly ground samples of fresh malt were substituted for that sent. The digestion with acid was carried on by putting the flasks in a box well supplied with hot steam and maintaining them at or near the temperature of boiling water for two and one half hours. Each flask was supplied with an open tube about three feet long, most of the tube being above the steam box. The precipitated copper was collected on asbestos in glass tubes and reduced in hydrogen.

W. H. Heileman, Washington.—The moisture determinations were made by the regular association method. The samples were extracted with ether previous to making the starch determinations. The copper was weighed as metallic copper, having been deposited electrolytically from a dilute nitric acid solution. The malt solutions were made for each determination. The malt was standardized by the same processes as those used in estimating the starch. The malt solutions were always fresh and of uniform strength. After washing the samples with alcohol, as directed, they were dried each time on the water bath, to entirely free them from alcohol, and were afterwards immersed in a boiling water bath with 50 cc of water, as directed. I found difficulty in knowing when the diastase had converted all the starch. Forty cc of malt solution were used for sample No. 1 and 20 cc for sample No. 2.

H. C. Sherman, Columbia University, N. Y.—In all cases after treating with malt solution until the starch reaction had disappeared, the solution was boiled and again tested for starch. If the latter were found, the treatment with malt extract was repeated.

S. S. Munson, Michigan.—With the diastase method for starch a fresh extract was made up for each determination, as at the beginning it was found that the extract

was ineffective after having stood two days. This method gave results that duplicated very closely in both cases. In sample No. 2 there was no reaction with iodin after thirty minutes, whilst sample No. 1 required about ninety minutes to completely convert the starch

DISCUSSION.

An examination of the data as calculated to dry matter reveals as great a variation from the mean as was exhibited in last year's work. This variation may be at least in part ascribed to the difference in the amount of moisture as found and used for calculating the results of the basis of dry matter. The following table contains these results:

TABLE 2.—*Moisture determinations.*

Analyst.	No. 1.	No. 2.	No. 3.
	P. ct.	*P. ct.*	*P. ct.*
E. B. Holland, Mass. Exp. Station	9.68	7.90	7.11
C. D. Howard, N. H. Exp. Station	8.59	6.79	.....
C. H. Jones, Vt. Exp. Station	9.43	7.35	.....
G. L. Teller and J. F. Moore, Ark. Exp. Station	12.37	10.27	9.03
W. H. Heileman, Wash. Exp. Station	9.66	7.47	.....
H. J. Patterson, Md. Exp. Station	10.20	7.89	7.33
F. S. Shiver, S. C. Exp. Station	9.26	7.40	6.63
H. C. Sherman, Columbia University, N. Y.	10.50	8.32	.....
O. C. Beck, Columbia University, N. Y.	.....	8.37	.....
H. Fisher, Columbia University, N. Y.	.....	8.40	.....
R. G. Wright, Columbia University, N. Y.	9.66	.....	.....
S. S. Munson, Mich. Exp. Station	11.30	9.10	8.05
W. H. Krug, U. S. Dept. of Agr.	9.91	8.42	7.67

The influence which such differences may exert is most strikingly shown in the results obtained by Mr. Jones and by Messrs. Teller and Moore, where the data given for the original material agree very satisfactorily, while the same results calculated to dry matter vary by 2.67 per cent.

The direct results obtained are, with the exception of the figures which have been excluded from the averages, quite satisfactory. The most plausible explanation for the low figures obtained in some cases is that the gelatinized starch had not been completely dissolved by the diastase, and therefore escaped the final inversion. Lintner and others have shown that the disappearance of the iodin reaction in the solution can not be accepted as proof of the total conversion of the gelatinized starch into soluble products, and German chemists determine this point by a microscopic examination of the residue. This is without doubt much more rational and certain, since it is but logical to assume that the starch suspended in the liquid is more rapidly and easily converted by the diastase than that which is partially or wholly surrounded by cell walls. It seems to me that we may here seek one of the causes of the deviations which have heretofore characterized the results obtained by this method, and that this source of error should be removed by determining the total conversion of the starch by means of a microscopic examination of the residue. This is easily done, and requires but a moment.

Another source of error may be sought in the solubility of the pentosans when the material is treated by the diastase method. König[1] first took up this subject, and published a number of results which showed that these bodies were partially dissolved. The matter was then investigated by Dr. Wiley and myself,[2] our data

[1] Landw. Versuchsstat, 48 (1896), 81.

[2] Journ. Am. Chem. Soc., 20, 4, 266.

indicating that only small quantities of the pentosans were soluble. Lintner[1] subsequently published an article on the determination of starch in cereals, in which he states that he finds the pentosans to be appreciably dissolved, but ascribes this rather to the action of the boiling water than to hydrolysis by the diastase. An analysis of barley is given, there being found 63.66 per cent of starch. The solution contained 2.84 per cent of pentosans (less pentosans present in the malt extract), giving as the true starch content 60.82 per cent. The same material was then inverted directly with acid according to the Sachsse method, the total reduction in terms of starch being 70.81 per cent. The solution contained 9.81 per cent pentosans, giving as the starch content 61 per cent. On account of the difference in the reducing power of the pentoses and dextrose these figures do not represent the true amount of starch present. The author assumes this to be slight, however, as he found that when a solution containing dextrose and the sugars obtained from the pentosans was fermented the unfermentable residue had a reducing power of 5.13 per cent in terms of starch, and the pentosans were found to be 5.19 per cent. He therefore considers it admissible to deduct the pentosans from the total reducing power stated in terms of starch.

Accepting the above as true, it can be easily understood that any variation in the time of treatment with diastase would result in the solution of different amounts of pentosans, and would thereby influence the final result very materially. The following results, which I obtained by a modification of the diastase method in which the gelatinized material was treated one hour at 50° with 20 cc of malt extract, boiled thoroughly, cooled, and again treated for an hour at 50° with 20 cc of malt extract, when starch could not be detected in the residue, indicate that this actually occurs:

TABLE 3.—*Results obtained with modified diastase method.*

Sample No.	Starch in air-dry material.
	Per cent.
1	67.29
2	13.33

A comparison of these data with the results which I obtained on the same materials by the provisional method, shows an increase of 1.84 per cent in the starch content of sample No. 1 and of 5.45 in the starch content of sample No. 2. This is just what we would expect if König and Lintner's statements are correct and the increased amount of pentosans which has been dissolved by the second boiling is more marked in sample No. 2, which contains a high percentage of pentosans as compared with sample No. 1. I am, however, not prepared to accept this explanation as final, since it does not seem to me to be based on sufficient analytical data.

The use of an increased quantity of malt extract gives rise to another error in the final result. Wroblewski has recently shown that diastase consists of a proteid combined with araban, the mother substance of arabinose. The error which is caused in this manner is necessarily small when the provisional method is adhered to, but increases with the amount of malt extract used.

Slight differences in the final results are no doubt caused by the fact that the copper was not weighed in the metallic state in all cases. In connection with this subject I wish to call attention to a new method of reducing the oxid which has been devised by Breshus.[2]

The cuprous oxid is collected on a filter, washed thoroughly, the filter partially dried between filter paper, transferred to a small weighed platinum dish, heated to

[1] Zeitsch. angew. Chem., 1898, 725.

[2] Neue Ztsch. Rübenzuck.-Ind., 41, 1, 4.

remove the water, and ignited. The dish is previously prepared by heating it to ignition, adding 1 cc of methyl alcohol drop by drop, allowing it to cool partially, and placing it in the balance case. When the filter has burned, the mass of cupric and cuprous oxids is moved to one side as much as possible, and the dish is covered with a lid, which has an opening in the center with depressed edges. It is heated to bright redness, and 1 cc of methyl alcohol is run in drop by drop. The flame is then removed and another cc run in. The vapors of methyl alcohol and formaldehyd are ignited, and as soon as they cease burning the cover is removed and the dish placed in the balance case to be weighed when cold. The operation must be repeated when any doubt exists with reference to complete reduction. Andrlik and Hraníčka[1] have suggested a modification of this method, in which they use a Gooch crucible, prepared as follows: The bottom is covered with asbestos, on which is placed a perforated platinum disk with a short platinum wire to facilitate its removal. This is in turn covered with a layer of asbestos. The double filter thus formed is washed with alcohol and the crucible dried and weighed. The cuprous oxid is collected and washed with hot water and alcohol. The crucible is then placed in a larger solid crucible, which is slowly heated until the Gooch crucible is at a red heat. It is then immediately transferred to a small beaker, the bottom of which is protected by means of copper gauze, and which contains some methyl alcohol. The beaker is covered with a watch glass. The crucible is removed when cold, dried for ten minutes in a water oven, and weighed. Comparative determinations have shown this method to be practically as accurate as the asbestos-tube method.

RECOMMENDATIONS WITH REFERENCE TO THE DIASTASE METHOD

It is recommended that—

(*a*) The quantity of material to be used be changed to 3 grams.

(*b*) In the first line the words "with ether, bring the extracted residue onto a filter or into a Gooch crucible, and" be stricken out, and the words "on a hardened filter, with five successive portions of 10 cc of ether," be substituted.

(*c*) In the sixth line and following, the words "until the solution no longer gives the starch reaction" be stricken out, and the words "until a microscopic examination of the residue with iodin reveals no starch" be substituted.

(*d*) That in the tenth line the word "exactly" be changed to "nearly," and the words "avoiding excess" be stricken out.

SUGGESTION.

It is suggested that the solubility of the pentosans be made the subject of further study in connection with the diastase method.

II. The Estimation of Pentosans.

In accordance with the suggestion contained in the report of the referee for 1897 the work consisted of a further comparison of the phenylhydrazin and phloroglucin methods. Both methods have been described fully in the last Proceedings of the Association.

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[1] Zeitsch. Ver. Rübenzuck.-Ind. deutsch. Reichs, 47, 1077.

The following table contains the results obtained:

TABLE 4.—*Comparative results with phenylhydrazin and phloroglucin methods.*

Analyst.	Barley.				Bran.			
	Original material.		Calculated to dry matter.		Original material.		Calculated to dry matter.	
	Phenylhydrazin method.	Phloroglucin method.	Phenylhydrazin method.	Phloroglucin method.	Phenylhydrazin method.	Phloroglucin method.	Phenylhydrazin method.	Phloroglucin method.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
E. B. Holland, Mass. Exp. Station	3.08	4.34	3.41	4.81	25.77	26.85	27.98	29.15
C. D. Howard, N. H. Exp. Station	.....	3.92	.....	4.29	.....	27.80	.....	29.83
Teller and Moore, Ark. Exp. Station	.....	5.15	.....	5.88	.....	27.80	.....	30.98
H. J. Patterson, Md. Exp. Station	.....	2.95	.....	3.29	.....	23.88	.....	25.92
F. S. Shiver, S. C. Exp. Station	4.79	3.96	5.28	4.36	26.13	25.80	28.22	27.86
C. H. Jones, Vt. Exp. Station	.....	3.42	.....	3.78	.....	[1]19.67	.....	[1]21.23
S. S. Munson, Mich. Exp. Station	3.53	4.32	3.98	4.87	[1]19.28	24.42	[1]21.21	26.86
W. H. Krug, U. S. Dept. of Agr	3.89	4.42	4.32	4.90	25.02	26.37	27.32	28.79
Averages	3.82	4.06	4.25	4.52	25.64	26.13	27.84	28.48

[1] Excluded from the averages.

NOTES ON THE ESTIMATION OF PENTOSANS.

H. J. Patterson, Maryland.—I found it impossible to obtain constant weight by drying from three to four hours. With sample No. 1 it required about twice that time, and with sample No. 2, I dried for ten hours.

F. S. Shirer, South Carolina.—In regard to the determination of the pentosans I would say that I allowed the solutions, after precipitation with the phenylhydrazin acetate, to stand twenty-four hours in all cases; then filtered, and washed in the usual way. The hydrazone precipitate was then dissolved in strong alcohol, the alcohol evaporated, and the residue dried at 50° to 60° C. to constant weight. I found the method as described in your circular unsatisfactory for two reasons, viz, the impossibility of removing all portions of the precipitate from the beaker, and also the incomplete precipitation of the hydrazone in the half hour specified.

C. H. Jones, Vermont.—If my pentosan results are low, it is probably due to the nature of the flame used in the distillation. This, I am just informed, should be so regulated that no charring of material occurs on the sides of the flask.

S. S. Munson, Michigan.—In both methods for determining pentosans it was impossible to obtain results that duplicated closely. The phloroglucin method invariably gave higher results than the phenylhydrazin method. With sample No. 1 the distillate usually gave a reaction with anilin acetate until about 400 cc had passed over; with No. 2 until about 450 cc had distilled over. In the phenylhydrazin method it was found necessary to let the solution stand for some time before filtering, in order to secure complete precipitation.

DISCUSSION.

The table shows that the phloroglucin method has almost uniformly given higher results, only one chemist reporting a higher percentage of pentosan by the phenylhydrazin method than by the phloroglucin method. This was obtained by allowing the solution to stand for twenty-four hours before filtering.

I have not been able to discover any cause for this uniform discrepancy, but am inclined to ascribe it to an incomplete removal of the furfurol-hydrazone from the beaker in which it has been precipitated. The precipitate is often of such a nature that when it has become attached to the sides of the beaker it is a matter of extreme difficulty to remove it completely. The crystals, when rubbed with a feather or glass rod, break up into a tarry mass, which persistently adheres to the glass and can not be detached. I do not think that the difference is due to an incomplete precipitation of the furfurol-hydrazone, as the phenylhydrazin method has been thoroughly studied with known amounts of furfurol and the factors which are used are based on the results of these studies.

The differences which occur between the results obtained with the phloroglucin method by the individual chemists may be due to several causes. Stift[1] has lately published an article on the determination of the pentosans by the phloroglucin method in which he shows that even the phloroglucin purissimum of Merck generally contains considerable quantities of diresorcin, and reports comparative analyses, made with impure and purified phloroglucin, which show deviations amounting with bran to as much as 0.28 per cent. According to Zeisel, the diresorcin may be removed by shaking with an equal volume of pure ether (20 grams of phloroglucin, 20 cc of ether) and washing the residue with ether until it no longer gives a violet color when dissolved in a few drops of acetic anhydrid heated and mixed with 1 to 2 drops of concentrated sulphuric acid. The phloroglucin dissolved by the ether can be recovered by distilling off the ether and purifying the residue, as before.

The influence which the presence of diresorcin exerts on the result has been the subject of considerable discussion, and Tollens and Krüger have found that small quantities do not impair the accuracy of the method. This is of some importance, as the purification is expensive. It is evident, however, that some limit should be placed on the amount of diresorcin present, especially when we consider that Councler found Merck's phloroglucin purissimum to contain from 7.59 to 14.63 per cent. Such amounts seriously vitiate the results, as the diresorcin is only difficultly soluble in hydrochloric acid of 1.06 specific gravity. Merck has now introduced a phloroglucin free from diresorcin, and I think that this or a similar preparation should always be used, and that, as a matter of precaution, the test above described should be applied.

Variations in the method of distillation cause errors, which, according to Stift, amount to as much as 1 per cent of furfurol. In a subsequent article Komers and Stift[2] recommended that the distillation be carried out at 140° to 160° in a bath of Rose's metal, at which temperature the distillate amounts to about 30 cc every ten minutes. I believe that the present method of distillation is deficient, as it is liable to cause a certain amount of charring or caramelization on the sides of the flask, which is quite difficult to prevent and will result in a variable loss. I have, unfortunately, not been able to make comparative tests on this point, but think it worthy of investigation.

I wish to call attention to the new factors which have been adopted by Krüger and Tollens for the conversion of the phloroglucid into furfurol and which are based on a series of determinations made with known amounts of furfurol and phloroglucin. They are as follows:

Phloroglucid (less than and up to 0.2 gram) ÷ 1.82 = furfurol.
Phloroglucid (from 0.2 to 0.3 gram) ÷ 1.895 = furfurol.
Phloroglucid (from 0.3 to 0.4 gram) ÷ 1.92 = furfurol.
Phloroglucid (above 0.4 gram) ÷ 1.93 = furfurol.

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[1] Oesterr. ungar. Zeitsch. Zuckerind. und Landw., 27, 19.
[2] Neue Zeitsch. Rübenzuck. Ind., 39, 7, 77.

I also wish to point out that the formulæ which are given for the conversion of the furfurol into pentosans do not agree with those given by Krüger and Tollens,[1] whose investigations led them to adopt the following for the phloroglucin method:

I. (Furfurol — 0.0104) × 1.68 = xylan.
II. (Furfurol — 0.0104) × 2.07 = araban.
III. (Furfurol — 0.0104) × 1.88 = pentosan.
IV. (Furfurol — 0.0104) × 1.91 = xylose.
V. (Furfurol — 0.0104) × 2.35 = arabinose.
VI. (Furfurol — 0.0104) × 2.13 = pentose.

The same authors have adopted the following formulæ for the phenylhydrazin method:

I. Hydrazone × 0.516 + 0.0104 = furfurol.
II. Hydrazone × 0.8681 = xylan.
III. Hydrazone × 1.0671 = araban.
IV. Hydrazone × 0.9676 = pentosan.
V. Hydrazone × 0.9865 = xylose.
VI. Hydrazone × 1.2126 = arabinose.
VII. Hydrazone × 1.0995 = pentose.

RECOMMENDATIONS.

It is recommended that—

(*a*) A phloroglucin free from diresorcin be specified for the precipitation of the furfurol.

(*b*) After the phloroglucid has been collected on the Gooch crucible, the action of the aspirator be continued for half an hour.

(*c*) The sentence "In case there is very little furfurol in the substance treated," etc., be stricken out.

(*d*) The time of drying be made three and a half to four hours.

(*e*) The following method for testing the phloroglucin be incorporated in the provisional method: Dissolve a small quantity of the phloroglucin in a few drops of acetic anhydrid, heat almost to boiling, and add a few drops of concentrated sulphuric acid. A violet color indicates the presence of diresorcin. A phloroglucin which gives more than a faint coloration must be rejected.

(*f*) That the factors adopted by Krüger and Tollens be substituted for those at present in use.

SUGGESTION.

It is suggested that the influence which variations in the method of distillation exert on the yield of furfurol be investigated.

III. The Estimation of Galactan.

The work consisted of a comparative study of the method proposed by Dr. Lindsey in the report for 1897. I subsequently found that Mr. Holland had determined the galactan both in the original material and after it had been extracted with ether. This appeared to be an important improvement, and I accordingly made a similar comparison and requested Mr. Teller, the associate referee, to do likewise.

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[1] Zeitsch. angew. Chem. 1896, pp. 36–46.

The following table contains the results:

TABLE 5.—*Results obtained with the galactan method.*

Analysts.	Original material.		Extracted material.	
	In original substance.	Calculated to dry matter.	In original substance.	Calculated to dry matter.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*
E. B. Holland, Mass. Exp. Station	8.14	8.76	9.33	10.04
G. L. Teller and J. F. Moore, Ark. Exp. Station	8.18	9.00	9.38	10.30
H. J. Patterson, Md. Exp. Station	[1]5.16	[1]5.57	.....	.....
F. S. Shiver, S. C. Exp. Station	9.33	9.99	.....	.....
S. S. Munson, Mich. Exp. Station	7.11	7.73	.....	.....
W. H. Krug, U. S. Dept. of Agr	7.67	8.31	9.01	9.76
Averages	8.09	8.76	9.24	10.03

[1] Excluded from the averages.

NOTES ON THE ESTIMATION OF GALACTAN.

G. L. Teller and J. F. Moore, Arkansas.—The weight of mucic acid obtained was much more constant for different determinations when the material was previously extracted with ether than when it was not.

F. S. Shiver, South Carolina.—The galactan determinations were not as satisfactory in my hands as they might be, quite large differences being found.

S. S. Munson, Michigan.—In the determination of galactan closely duplicating results were secured.

DISCUSSION.

It is seen that previous extraction with ether increases the percentage of galactan found quite materially and results in more uniform work. It is without doubt a valuable improvement and worthy of adoption.

RECOMMENDATION.

It is recommended that—

The provisional method for the estimation of galactan be amended so that the first line shall read, "Extract 3 grams of the finely powdered substance on a hardened filter with five successive portions of 10 cc of ether, bring the extracted residue into a beaker," etc.

An extended discussion of the report was engaged in by the members, with especial reference to the determination of carbohydrates and organic acids. It was pointed out that in the reports of the analyses of feeding stuffs the organic acids were usually omitted. It was further pointed out that if all the different constituents known already to exist in feeding stuffs be determined, the summation of the data of the analysis approximates very nearly 100 per cent. This is especially true of cereal grains, but in cases of fodders, hays, straws, etc., the discrepancies are still somewhat large.

The president then called for the report of the referee on food adulteration. In the absence of the referee, Mr. W. S. Sweetser, the secretary read his report:

REPORT ON FOOD ADULTERATION.

By W. S. Sweetser, *Referee.*

Gentlemen: Your referees decided that the time had come when some attention might be given profitably to the comparison of some of the laboratory methods employed in this branch of analytical work. The subject of starch determination in spices was selected, not because it is the most important determination made in the detection of adulteration, but because the subject seems to be so open to investigation and is one this association is investigating in other relations.

Invitations to cooperate in this work were sent out in the spring, but only a very few favorable replies were received. The work laid out was of a nature to require but a little time, yet very few of the chemists to whom these invitations were sent were in a position to undertake the work. Three lots of whole spices—nutmeg, Jamaica ginger, and Singapore pepper—were reduced to a very fine state in the Menker fodder mill. The samples were not sifted. They were tightly corked and sealed and sent to the different laboratories with the following directions:

I. Diastase Method.

starch.

Exhaust 3 grams of the substance with ether and treat the residue on a Gooch with 10 per cent alcohol till the soluble matter is extracted, wash with strong alcohol, bring the dried residue into a 250 cc flask with 50 cc of water, immerse flask in a boiling-water bath, stirring the contents constantly until the starch gelatinizes; cool to 55° C. and add 40 cc of malt extract and maintain this temperature until the solution no longer gives the starch reaction with iodin. After cooling make up to 250 cc, filter, and invert 200 cc in a 10 oz. flask with 20 cc of 25 per cent HCl, sp. gr. 1.125, connect with a reflux condenser and heat in a boiling-water bath for two hours, cool, and when milk-warm neutralize carefully with NaOH (avoiding an excess), clarify, if necessary, with 10 to 20 cc of alumina cream, and make up to 500 cc. Mix well, pour through a dry filter, and determine dextrose in an aliquot part by Allihn's method. Convert dextrose to starch by the factor 0.9.

Preparation of malt extract.—Digest 10 grams of fresh, finely ground malt over night at ordinary temperature with 200 cc of water and filter. Determine the amount of dextrose in a given quantity of the filtrate after boiling with acid, etc., as in the starch determination, and make the proper correction.

II. Direct Inversion.

starch.

Exhaust 3 grams of the substance with ether, treat the residue on a Gooch with 10 per cent alcohol, and then with strong alcohol; dry, transfer to a flask, add 200 cc of water and 20 cc HCl, sp. gr. 1.125, connect with a reflux condenser, and boil for four hours. Filter out the residue and accurately neutralize the filtrate with NaOH. Clarify, if necessary, with alumina cream. Mix well, run through a dry filter, and determine dextrose in an aliquot part by Allihn's method. Convert dextrose to starch by the factor 0.9.

In addition to the starch determinations by these two methods, the analysts were requested to determine volatile oil by the method considered preferable and to report the method in detail. The following results and notes were received:

Analyst.	Nutmeg.					Ginger.					Pepper.				
	Moisture.	Volatile oil.	Fixed oil.	Starch, diastase.	Starch, direct inversion.	Moisture.	Volatile oil.	Fixed oil.	Starch, diastase.	Starch, direct inversion.	Moisture.	Volatile oil.	Fixed oil.	Starch, diastase.	Starch, direct inversion.
C. D. Howard, Durham, N. H	*P. ct.* ----	*P. ct.* 3.03	*P. ct.* 35.10	*P. ct.* 20.72	*P. ct.* 25.63	*P. ct.* -----	*P. ct.* 1.47	*P. ct.* 3.12	*P. ct.* 51.97	*P. ct.* 57.57	*P. ct.* -----	*P. ct.* 1.22	*P. ct.* 7.23	*P. ct.* 39.15	*P. ct.* 41.73
W. L. Mitchell, New Haven, Conn	----	3.87	38.29	-----	-----	-----	1.49	3.04	-----	-----	-----	.90	7.31	-----	-----
C. A. Browne, jr., State College, Pa	9.74	----	-----	25.13	25.46	11.39	----	----	54.04	58.24	10.93	----	----	38.21	43.45

NOTES.

C. D. Howard.—In the determination of volatile oils 3 grams were extracted from twenty-five to twenty-eight hours in a Johnson continuous extractor. The extract was transferred to a 50 cc beaker and exposed over night and then dried over H_2SO_4 and pumice stone to a constant weight, which was difficult to obtain. It was then dried a short time at 80° C. and finally two hours at 110° C. The loss was called volatile oils and the residue fixed oils. No difficulty was experienced with the starch work except in filtering the alcohol extract from the pepper.

W. L. Mitchell.—The method used for ether extract was that described by Richardson, Bulletin No. 13, Division of Chemistry. Two grams were extracted twenty hours with ether without previous drying. After allowing the ether, etc., to evaporate spontaneously in a capsule, the residue was dried twelve hours over H_2SO_4, thus giving the total extract. The loss after drying to a constant weight at 110° C. was called volatile oil and the residue non-volatile ether extract. A second extraction of ten hours gave the following per cents of non-volatile ether extract: Pepper, 0.15; nutmeg, 0.13; ginger, 0.11.

Dr. A. L. Winton gives the benefit of some of his experience along this line of work. He used twelve samples of pepper—black, white, shells, long, etc.—and treated each according to the two methods described above. His first difficulty was with the malt extract. Standing over night it invariably soured, owing to the warmth of the laboratory. It seemed necessary to fall back to the old plan of digesting three hours at 40° C., and using as soon as possible. In each case 4 grams of the washed pepper were digested four hours at 55° C., with 50 cc of the malt extract. The bulk of the liquid, which was 100 cc at the beginning of the digestion, 50 cc starch paste and 50 cc malt extract, was increased to 150 cc at the end of the third hour to remove the paste adhering to the sides. At the end of the fourth hour the solutions gave no blue with iodin, and the analyses were finished in the manner directed. Unfortunately, he found, when too late, that there was still undissolved starch in the solid part of the pepper, and the fact that the results were from 5 to 8 per cent lower than by the direct inversion was largely due to this cause. Time was too limited to follow the matter up before this meeting.

Fine grinding aids in the malt digestion, but renders the preliminary washing very difficult and so slow that fermentation is liable to set in. Then, too, there is danger of loss of starch mechanically, especially with those spices whose starch grains are very minute.

Longer digestion with the malt might be more effective, but there might be the possible danger of the malt beginning to ferment by protracted heating at 50° to 55° C.

Interrupted digestion is also suggested. This plan is to digest with a certain amount of malt extract for one hour, then boil, add more malt extract, after lowering to the proper temperature, and digest again. This process could be repeated. The boiling would probably render the starch more gelatinous and easily acted upon than the mere heating in a bath of boiling water. This plan of interrupted digestion seems worthy of some attention, especially with those spices which should not be ground very fine.

Your referee suggests that this subject receive further attention, and that these two methods of starch determination, with some modifications, be applied side by side to spices quite low in starch content, such as cayenne, cloves, and mustard.

In the examination of such material, a quick, accurate method for the determination of starch would be valuable.

The president called for papers on the subject. None having been presented, the discussion of the subject was declared to be in order.

Mr. W. D. Bigelow said he was much interested in this matter and would be glad to hear from the president of the association, who had lately done a great deal of work, on spices especially. The great object of the study, of course, is to enable one to judge of the purity of the spices offered for sale. These spices often have starch added as an adulterant, for instance, wheat starch. There should be some standard on a subject of this kind, and he understood that the president was engaged in work which might lead to the establishment of just standards for spices and condiments of all descriptions. The present methods of examination seem to be too tedious. Shorter and yet reasonably accurate methods should be devised, if possible. Perhaps it might be sufficient to make a hydrolysis of the spice and polarize it directly, or the diastase solution might be polarized directly. Such a method, while not at all accurate, might be satisfactory for sorting purposes.

The president said he was very full of this subject. He came from a State which was supposed to be the original home of the fabrication of nutmegs, and was naturally interested in spices and condiments of all kinds. He said he had samples at his hotel of spices and condiments which he would gladly bring up and show to the association, having collected a great number for analytical purposes.

The method in use by him was to determine the oils, ash, water, and alcohol extract, nitrogenous bodies, and in addition to this special microscopic examinations. A great many adulterations of spices were in the way of the use of exhausted spices, such as ginger. The microscope has no value in discriminating between exhausted and non-exhausted spices. In England the custom is to determine the cold-water extract, starch, etc. Full reports of the president's work will be found in later publications of the Agricultural Experiment Station of Connecticut, food adulteration department.

Mr. H. A. Huston called attention to the fact that the manufacturers and dealers should have their names on the packages of spices, so that they could be identified.

Mr. Bigelow called attention to the desirability of publishers of chemical books printing their advertisements on cards similar to those used for library purposes, so that these advertisements might be conveniently filed. The association voted to concur in Mr. Bigelow's suggestion.

The president begged leave to call the attention of the association to the death of one of its associate referees, namely, Dr. Charles P. Worcester, of Boston. Dr. Worcester was in ill health at the time he accepted the position of associate referee, but was so much interested in the work that he consented to do the best possible. Death, however, brought his career to an untimely end. The president asked the members, as a tribute to the memory of the late Dr. Worcester, to rise in their places.

The president appointed the following committee on nominations for offices for the next year: Messrs. Scovell, Ross, and Frear. The president then declared the convention adjourned until afternoon.

SATURDAY—AFTERNOON SESSION.

On the reassembling of the association the president called for the report of the committee on recommendations of referees. The committee presented its unanimous report in favor of the recommendations of the referee of foods and feed stuffs. The report was adopted.

The report on dairy products was then called for, and it was read by the referee, Mr. C. L. Penny, as follows:

REPORT ON DAIRY PRODUCTS.

By C. L. Penny, *Referee.*

The work on which assistance was asked from members of the association for this year is outlined in the following circular letter:

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

METHODS FOR THE ANALYSIS OF DAIRY PRODUCTS.

Your cooperation is requested in studying the following methods for the analysis of dairy products:

I.—*Volatile acids of butter fat.*

1. Compare the provisional method of saponification, using a reflux tube or condenser (Circular 4, p. 3), with the official method prescribing a closed flask.

2. Compare with the official method of saponification the Leffmann-Beam method, using, if possible, both fresh and rancid butter. (See Analyst, Vol. XVI, p. 153; Vol. XVII, p. 65 and p. 176, and Vol. XXI, p. 259.)

II.—*Casein in milk.*

1. Modification of provisional method 1. (Acetic acid method, Circular 4, p. 3.) "Carry the method through as described to the washing of the precipitate, then neutralize the filtrate as exactly as possible with caustic alkali; filter off any precipitate that may form and add it to the former precipitate for the nitrogen determination."

2. Alum method: "To 10 cc of milk add 50 cc of water at 40° C. Then add 1.5 cc of an alum solution saturated at 40° C. or higher. If the precipitate fail to settle promptly, add half a cubic centimeter more of the alum solution, filter, wash with water, and use Kjeldahl method for nitrogen."

III.—*Albumin in milk.*

Modification of provisional method: "To the filtrate from the casein, according to either of the above methods, add 10 cc of strong tannin solution; heat, filter, wash with water, and determine nitrogen."

The proposed work on milk is suggested by the last referee, Dr. Van Slyke. Samples of milk will be sent to you for the casein and albumin if you are willing to cooperate in that part of the work.

C. L. Penny, *Referee.*

Newark, Del., *April 29, 1898.*

I. VOLATILE ACIDS OF BUTTER FAT.

1. Condenser versus Closed Flask.

Your referee was directed to investigate the propriety of using a flask attached to a reflux condenser for the saponification of fats in place of the closed flask. Dr. Weems, of the Iowa Station, found a Reichert number of 20.65 by the present method with closed flask, and 20.75 with condenser, each result being a mean of two determinations. Mr. C. H. Jones, of the Vermont Experiment Station, found, working with four different samples, higher results with the condenser in the case of two, viz, 0.15 cc and 0.31 cc higher, respectively, and in the case of the other two, higher results with the closed flask, viz, 0.31 cc and 0.38 cc higher, respectively. The mean of the four is 0.03 cc higher with the closed flask, and this must be regarded as complete concordance. It will appear from this that nothing is lost by the use of the condenser, as the average results agree perfectly. As the ethyl butyrate, whose escape is feared, boils at about 120, the condenser would seem to fulfill the extreme of precaution, and in fact to be more certain than the secure closing of a flask to withstand an internal pressure of over an atmosphere. It is probable that most of the experience on volatile acids has been gotten with the use of a condenser, as that seems to be the method in more general use. It is questionable, however, whether the open flask on a water bath, as prescribed by König in his Agricultural Analysis (Landwirtschaftliche v. Gewerb. Stoffe, 403), is not entirely too incautious. It would seem from the results given above and from general experience to be unquestionably as safe to use a condenser as a closed flask. I have no hesitation in recommending to the association the adoption of this provisional method as official.

2. Leffmann-Beam Saponification Method versus the Official Reichert-Wollney Method.

About six years ago the attention of the association was directed to the Leffmann-Beam saponification method. In the light of some analytical results presented at that time no favorable recommendation touching its adoption was made, and beyond discussion no action was taken. Since then no official report has been made to the association on the subject.

This method, as is well known, consists in heating in an open flask over a direct flame the butter fat with a solution of caustic soda in glycerol. The saponification takes place very rapidly and at a temperature considerably above 100°; in fact, it appears to be almost instantaneous as compared with the Reichert-Wollney method. Water is added at once to the soap thus formed, then dilute sulphuric acid, when the distillation is begun without previous melting of the fatty acids. Thereafter everything is as in the official method.

The advantages of the glycerol method are so obvious to anyone who has tried it that little need be said. When it is remembered that the official method advises one hour for saponification proper, and for solution of free fatty acids a time varying from a few minutes to several hours, the saving of time by the glycerol method is enormous, reducing to five minutes at most what requires one or more hours. It is doubtful if any other analytical method have been devised within recent years that so greatly abridges a process as does this one. So easy is it in the execution, furthermore, that the only question as to its precedence of all rivals is as to the results obtained.

It is much to be regretted that fuller original experimental data can not be laid before the association, but I have available only the results of Dr. Weems and my own.

Dr. Weems found 20.21 cc decinormal soda as a mean of two determinations for 5 grams of fat, using 20 cc of a 5 per cent solution of soda in glycerol, and again 20.32 cc decinormal soda using 10 cc of a 12 per cent glycerol solution. The official method gave as a mean of two determinations 20.65 cc decinormal baryta. Apparently two different standard solutions were used for the titration, soda for the new

method and baryta for the old. The discrepancy is 0.39 cc less by the glycerol method.

My own results are as follows:

On a single sample of fresh butter four repetitions by the official method averaged 28.80 cc and three by the Leffmann-Beam 28.63 cc, a shortage for the latter of 0.17 cc. In a series of comparative tests on 10 different samples from ten to fourteen months old, and consequently rancid, the official method averaged 0.78 cc above the Leffmann-Beam. In 14 similar comparisons of 11 samples all less than a month old, the official method averaged 0.49 cc above the other. Thus the Leffmann-Beam method seems to give slightly lower results than the official method, on a general average of the above figures 0.35 cc for fresh samples and, as just stated, about 0.78 for rancid samples.

Two questions may with propriety be asked: First, is this shortage an error? and, second, if it be an error, is it a material one? The Leffmann-Beam method requires the sample and reagent to be heated considerably above 100° (as judged by the explosion of the refluent drops of water) before the reaction sets in. During this raising of the temperature there is doubtless a certain loss of free volatile acids. Is this a defect or a merit? By the official method such escape can not occur except during the prolonged heating of the original butter sample "at about 60° for two or three hours" when such escape does unquestionably occur, especially with rancid samples. What are these free volatile acids? Are they on the one hand original constituents of the pure natural butter fat or liberated from the same, or, on the other hand, are they fermentation products of non-fatty bodies left in butter, as casein, milk sugar, and lactic acid? If these free volatile acids have been liberated from butyrin with separation of glycerol, they ought to be counted in the final distillate. If they have been formed by fermentation from lactic acid or milk sugar they are as much foreign to butter fat as is cottonseed oil, and they ought to have no place in the distinction of butter fat from other fats. Or, to apply the question directly to the end in view, in the sum total of volatile acids taken as characteristic of butter fat should the free volatile acids of the original sample be included? There are arguments on both sides. It has been found that butter after a long time, as several years, loses a little of its total volatile acids, a fact seeming to indicate that everything volatile should be counted; whereas there is doubtless a shorter time after making when the sum of volatile acids is increased by fermentation products of the sugar and lactic acid, bodies less stable than the fats and more readily split up. To take evidence from practical life, the readiness with which poorly worked butter becomes rancid seems to emphasize the fact of an accession of volatile fatty acids from a non-fatty source. As a choice between uncertainties, since to the analyst the age and other conditions of a sample are supposed to be unknown, I am of the opinion that the Reichert number should be understood to represent combined volatile fatty acids, with exclusion of the free. Should this view obtain, the slight shortage by the Leffmann-Beam method would not be considered a defect. It would seem that the official method ought to aim at one thing or the other, either to count all of the volatile acids, free and combined, or to count only the combined. At present it does neither. The melting of the sample at 60° for two or three hours is, of course, attended with some loss of free acids, as may easily be detected by the sense of smell. If it be designed to include such acids as characteristic of butter fat, then the preliminary official method of treating the butter sample calls for revision.

As to the second question: If any deficiency as compared with the official results be assumed an error, is it a material one? It must be remembered in considering the Reichert distillation that we are dealing with something practically of high utility but logically incomplete and fallacious, in that the distillate obtained is but a fraction of the characteristic body present in the sample, and furthermore, a fraction variously estimated, under different manipulation, as low as 80 per cent and as high as 87 per cent, so that the distillation alone from the same soap might vary by one-twelfth or by 2 cc or more. (See Analyst XX, 218, and Allen, Organic Anal., II, 45.)

If there be any mode of reasoning repugnant to the true scientific spirit, it is this inference from an unknown part to the whole. The only justification in this instance is the lack of something better. The practical end aimed at is attained with as much certainty as fluctuations in natural butter fat will permit, and that, in default of something better, is enough.

It must be clear that the vulnerable point of the entire Reichert method is the distillation, not the saponification, the uncertainty as to the precise fraction that is distilled over. This uncertainty has been reduced so far as possible by the prescription of volume of liquid to be boiled, volume of distillate, time of boiling, etc. But there still remain unfixed conditions and practically unfixable, in the atmospheric pressure, shape of flask, etc., so that after the soap has been formed, the acids liberated and melted, if the solution were given to different chemists to distill there might be expected results varying as widely as any we find in our comparative tests. These differences between comparative tests are, however, slight. Let anyone compare them with the results obtained by different chemists on the same sample of phosphoric acid, for example, and it will be seen that this method, incomplete and fractional as it is, compares favorably in concordance with some of the oldest and best tried methods of analytical chemistry.

It must furthermore be borne in mind that the present application of the volatile acids determination is to distinguish genuine butter fat from other fats. When it is remembered that the Reichert number for genuine butter fat has been found to vary as much as 2 to 1, from 34 cc decinormal alkali to less than 15 cc, it will be clear that a close discrimination between butter fat and other fat, or a quantitative determination of a mixture of each, must be within wide limits of possible variations, so wide, in fact, that the discrepancies here presented are hardly as great in practical effect as the errors of a good balance, i. e., they are nothing. What rational chemist would dare to say that a sample giving a Reichert number of 20 is genuine and another giving one of 19 is adulterated? Such arbitrary decisions have been made, it is true. With the confidence that inexperience alone gives, it was held that any result below a fixed Reichert number should indicate adulteration, and the proportion of the adulteration was to be determined as certainly as by the rule of three. But widening experience with the vagaries of nature in the making of butter fat reduced this number from 28 to 26, to 24, even to 20. Unfortunately, Spallanzani found samples below 15. These are, for the sake of the analyst's assurance, regarded as abnormal (though how he shall say that his unknown sample may not also be abnormal, but genuine, is not clear). At all events the quantitative result of the Reichert method aims primarily at a qualitative conclusion, the question of more or less being a minor one. In this it differs from almost all other analytical processes. A judgment passed upon the general Reichert method that ignores this vital distinction is erroneous. It is a mistake to assume that analytical fluctuations have the significance here that they have in phosphoric acid determination for example, where analyses fix money values in strict proportion, though, as was said, the method under discussion will bear the comparison. The consideration of the value of a method ought to regard its application as well as its inherent qualities. In the light of all that can be ascertained I do not hesitate to recommend to the association for its unqualified adoption the Leffmann-Beam saponification method.

II. CASEIN IN MILK.

Two modifications of the present method have been proposed: First, the neutralization of the acetic acid filtrate, in order to obtain a second precipitate which is to be added to the main precipitate; second, the precipitation of casein with a saturated solution of alum instead of acetic or other acid. The comparative analytical results which follow are somewhat fragmentary, in that the several analysts have not covered the same ground, yet some inference may be drawn.

CASEIN AND ALBUMIN IN MILK.

Analyst.	Total N × 6¼.	Casein.			Albumin.		
		Provisional method.	Acetic filtrate neutralized.	Alum method.	Provisional method filtrate.	Neutralized filtrate.	Alum filtrate.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
C. H. Jones, Vt. Exp. Station.	3.37	.....	2.78	2.73	.....	0.40	0.59½
	3.41	.....	2.79	2.69	.....	.39	.62
	3.41	.....	2.76	2.66	.....	.38	.63
	3.40	.....	2.78	2.69	.....	.39	.61½
I. J. Meade, Iowa Exp. Station	.....	[1]1.62	.....	[1]1.94	0.66	.....	.60
J. A. Le Clerc, Geneva, N. Y., Exp. Station.	3.26	2.68	2.80	2.68	.....	.....	.....
	.....	2.69	2.81	2.69	.....	.....	.....
	.....	2.75	2.81	2.74	.....	.40	.44
	.....	2.68	2.80	2.77	.....	.42	.44
	.....	2.75	2.88	2.83	.....	.43	.45
	.....	2.71	2.82	2.74	.....	.42	.44
C. L. Penny, Del. Exp. Station	.....	2.64	.....	2.61	.....	.....	.....
	.....	2.59	.....	2.60	.50½	.....	.....
	.....	2.59	.....	2.61	.55	.....	.....
	.....	2.61	.....	2.61	.53	.....	.....
G. E. Patrick and T. C. Trescot, U. S. Dept. of Agr.	.....	.....	2.56	2.44	.....	.46	.56
	.....	.....	[2]3.21	2.44	.....	.45	.56
	.....	.....	[2]3.25	2.44	.....	.45	.56
	.....	.....	2.44	2.50	.....	.....	.58
	.....	.....	2.56	2.44	.....	.47	.56
	.....	.....	2.52	2.45	.....	.46	.564

[1] Presumably an arithmetical error, the percentage of nitrogen being unknown to the referee.

[2] These two results, evidently erroneous, were inexplicable to the analysts and were therefore reported; but they are not included in the average.

As to the modification of the acetic method (consisting in neutralizing the acid filtrate with caustic alkali and recovering any precipitate that may have formed) Mr. Le Clerc finds an increase of 0.11 per cent while I could not detect a second precipitate. The alum method is found by Messrs. Jones and Le Clerc to give from 0.08 to 0.09 per cent less than the modified acetic method, and by Messrs. Patrick and Trescut 0.07 per cent less, while I find equal results.

III. ALBUMIN IN MILK.

As to the determination of albumin by means of a strong tannin solution, the results range from 0.66 by Mr. Meade to 0.39 per cent by Mr. Jones, discrepancies influenced somewhat, though not wholly caused, by the nature of the filtrate from which the tannin precipitation was made. Without further comment on the results and without recommendation I offer a suggestion for future investigation, viz: That the total nitrogen be determined directly; that a relatively large quantity of milk, as 75 grams, be diluted about fivefold and be treated according to the alum method in liter flask and made up to the mark; in an aliquot of the filtrate from this let the nitrogen be determined and (multiplied by 6.25) let it be counted as albumin; let the casein be found by difference. This would dispense with the washing of the casein, which is a tedious process. If there be any nitrogenous body in milk properly included neither in that which acetic acid and alum precipitate nor in that which

tannin precipitates, it must be present in minute proportion; it is hard to see what advantage can accrue from so extended a subdivision of the proteids in milk, even should the actual existence of a number of such bodies other than casein and albumin seem to justify that step. By far the readiest way, then, of determining the casein and albumin would seem to be to count for albumin all proteids that the alum does not precipitate and to deduct this from the total sum of proteids. By this method there would be but a single precipitation, no washing of a precipitate, and two determinations of nitrogen, the total and the albuminous. The results of Mr. Le Clerc, added, show the casein plus albumin to be within 0.02 per cent and 0.08 per cent of the total proteids; Mr. Jones finds the sum of casein and albumin to fall 0.23 per cent and 0.10 per cent short of total proteids, a result due to his higher figure for total nitrogen rather than to lower figures for casein and albumin.

RECOMMENDATIONS.

(1) That after the last line on page 27 of the Methods the following be inserted: "(a_2) The fat prepared as above may also be weighed into the saponification flask from a weighing tube marked to contain 5.75 cc."

(2) That the provisional method of saponification, given in the fifth paragraph of page 3, Circular No. 4, be made optional.

(3) That after the first full paragraph on page 29 of the Methods the following be inserted:

LEFFMANN-BEAM METHOD.

(1) *Reagents.*

(*a*) GLYCEROL-SODA SOLUTION.—Add 20 cc of a 50 per cent solution of sodium hydroxid to 180 cc pure concentrated glycerol. The soda must be as free from carbonate as possible.

(*b*) SULPHURIC ACID.—Make up 200 cc of concentrated sulphuric acid to 1 liter.

(2) *Saponification and distillation.*

About 5 grams of fat are weighed into a flask and 20 cc of the glycerol-soda solution added. The flask is then heated over a Bunsen burner until complete saponification takes place, as evidenced by the mixture becoming perfectly clear, the whole operation requiring less than five minutes. The soap is then dissolved in 135 cc of water, preferably previously boiled, the water being added at first drop by drop, to prevent foaming; 5 cc of the dilute sulphuric acid are added and the distillation is carried on at once, as usual, without the previous melting of the free fatty acids.

At the conclusion of the reading of the report papers were called for, and none being presented, discussion followed on the subject of the melting points of fatty acids before distillation. It was the general consensus of opinion that it was not necessary to wait for a complete fusion of the fatty acids before beginning the distillation, but that the distillation might begin as soon as the acid was added and the soaps entirely decomposed.

Attention was called by Mr. Scovell to the fact that if only just enough acid be added to neutralize the alkali it requires a longer time to melt the separated fatty acids, while the addition of an excess of acid facilitates the fusion.

It was stated by Mr. Hand that for the ordinary quantities employed 25 cc are usually enough.

It was stated by Mr. Penny that the quantity of acid and alkali employed in the Leffmann-Beam method was the same as with ordinary procedure.

The committee on recommendations here presented its report in favor of the recommendations of the referee on dairy products. The report was adopted.

The PRESIDENT. The hour of 2 o'clock having come, the special order is the election of officers for the ensuing year. I therefore call upon the committee on nominations for a report.

Mr. SCOVELL. Mr. President, your committee on nominations begs to submit the following recommendations:

For president, Mr. R. C. Kedzie, of Michigan; for vice-president, Mr. B. W. Kilgore, of Mississippi; for secretary, Mr. H. W. Wiley, of Washington; for additional members of the executive committee, Mr. H. J. Wheeler, of Rhode Island; Mr. B. B. Ross, of Alabama; Mr. William Frear, of Pennsylvania.

The secretary was instructed to cast the ballot of the association for the nominees of the committee. The secretary reported that he had cast the unanimous ballot of the association in favor of the candidates named.

The report of the referee on sugar was next called for. The referee, Mr. R. S. Hiltner, then presented the following report:

REPORT ON SUGAR ANALYSIS.

By R. S. HILTNER, *Referee.*

As there has been no work done by the association on sugar analysis and no official report on the subject for the last two years, it is possible that many members have lost track of the lines of work in progress previous to that time. On this account, before discussing the work of the present year, a brief résumé of the work already done on the subject may not be out of place.

As gleaned from published reports of proceedings and from correspondence with our secretary, Dr. Wiley, the work that has been carried on may be summed up as follows:

At the fifth annual meeting the first report of the committee on sugar was read. The committee simply suggested general methods for the analysis of juices, molasses, etc. There was no formal report on sugar at the sixth convention, but a paper was read by Mr. B. B. Ross, in which he described experiments in the redetermination of Clerget's constant. At Dr. Wiley's suggestion, the methods of the Louisiana Sugar Association were adopted provisionally. Subsequent to the sixth meeting the work of the association has been confined largely to the methods of desiccation of various products. The "double polarization" methods and determinations of ash have also received some attention.

It is but a truism to state that sugar analysis is a broad field and that there yet remains much valuable work to be done in the investigation of both beet and cane products. The real difficulty in the way of rapid progress in this work is not a scarcity of material nor of subject matter, but rather a lack of time. Unfortunately, few members of the association can spare the time required by such work.

Of late years the demand upon the time of the station chemist has been so varied and the pressure of routine work so great as to considerably retard aggressive work for the association, and consequently the interest in many of the branches has considerably lagged. The effect of this has been keenly felt in arranging the work on sugar analysis this year. Of the 55 stations only 16 chemists offered to assist in this work, and of these only 4 reported results. The almost unanimous reason for declining to take part in the investigation was "lack of time." This state of affairs is greatly

to be regretted, because the value of our association work depends, to a very large degree, upon the number included in the averages.

For the work on sugars for the year the chemists of the association were asked to cooperate in analyzing some sugar-beet products. This work was undertaken on the suggestion of several chemists interested in sugar analysis. In view of the probable increase in the extent of the beet-sugar industry in the Northern and Western States it was thought desirable that the association should adopt at an early date analytical methods applicable to such products.

To be sure, many of our official methods that apply to cane-sugar products apply equally well to sugar-beet products. Moreover, the same difficulties in the way of obtaining accurate concordant results are met with in both classes of material. The difference, however, in chemical composition of the juices of the two sugar-bearing plants, cane and beet, and the difference in the processes used in the manufacture of sugar from them, result in factory products and by-products that differ widely in composition and characteristics, both chemical and physical. For example, the molasses from cane-sugar factories contains sucrose (about 35 per cent), reducing sugars (about 10 per cent), but no raffinose, while, on the other hand, the average sugar-beet molasses contains about 50 per cent sucrose and 0.4 per cent to 3 per cent raffinose, but little or no reducing sugars. This, of course, necessitates different analytical methods.

The material selected for the experimental work was a sample of sugar-beet molasses furnished by Supt. W. H. Baird, of the Norfolk Beet Sugar Factory. This molasses, a dark, viscid liquid, is rich in sucrose, contains a large percentage of ash and organic matter not sugar, as well as raffinose and a small amount of reducing sugar. It was considered, therefore, that this substance would represent in a general way the beet-sugar by-products, and that also, because of its complex composition, it would illustrate the difficulties in the way of obtaining accurate results.

The suggestions made by the referee for the analysis of the molasses were as follows:

I.—*Determination of water and total solids.*

(*a*) Determine per cent of water by the Carr and Sanborn method, which has been adopted, provisionally, by the association.

The drying may be conducted in a water oven at ordinary pressure or in vacuo.

(*b*) In a flat-bottom dish place 6 or 7 grams of pure quartz sand and a short stirring rod. Dry thoroughly, cool in a desiccator, and weigh. Then add 3 or 4 grams of the molasses, mix with the sand, and dry at the temperature of boiling water for eight to ten hours. Stir at intervals of an hour, then cool in a desiccator and weigh. Stir, heat again in the water oven for an hour, cool, and weigh. Repeat heating and weighing until loss of water in an hour is not greater than 3 mg.

This sample may be used for the determination of ash by the Alberti-Hempel method. (See Method II (*d*) under "Ash.")

The sand used should be pure quartz. It should be digested with strong HCl, washed, dried and ignited, and kept in a stoppered bottle.

II.—*Determination of ash.*

(*a*) Weigh 5 to 7 grams of molasses in a flat-bottom porcelain or platinum dish. Saturate with concentrated sulphuric acid, heat cautiously until the acid is driven off and until intumescence ceases, then ignite in a muffle at low redness. Subtract one-tenth of the weight of the ash and calculate per cent.

(*b*) Place in a porcelain dish 6 or 7 grams fine quartz sand and a short stirring rod, heat to redness, cool in a desiccator, and weigh. Then weigh into the dish 3 or 4 grams of the molasses, mix thoroughly with the sand, heat carefully at first to expel volatile matter. Place in a muffle and ignite at a low red heat.

(*c*) In a platinum ash dish heat 5 to 7 grams of the sample over a flame until intumescence ceases, then ignite in a muffle at low redness until the ash obtained is white, or nearly so.

(*d*) (Modification of the above method). Weigh a sample of the molasses, add a little pure olive oil (enough to form a thin layer), heat, and ignite as above. The oil serves to prevent excessive frothing; it must be free from non-volatile matter.

(*e*) If time permit, please determine ash also by methods (2) and (6), page 49, Bulletin 46, Division of Chemistry.

In each case after weighing the ash, heat again in the muffle for fifteen minutes and weigh. Repeat if the weight be not constant.

III.—*Determination of reducing sugars.*

(*a*) For the determination of reducing sugars follow the "gravimetric method" of the Association of Official Agricultural Chemists. (1 (*a*), page 51, Bulletin 46, Division of Chemistry.) Filter and wash the precipitated cuprous oxid in a Gooch crucible as directed under (*b*), page 56, Bulletin 46. Then determine the copper in nitric acid solution by the electrolytic method. (See (*d*), page 56.)

(*b*) In another sample determine the reduced copper by the "permanganate" method. (See (*e*), page 56.)

In each case state the weight of molasses employed, the weight of copper obtained, and the per cent of invert sugar.

IV.—*Determination of sucrose and raffinose.*

Dissolve 26.048 grams of the molasses in water, transfer to a 100 cc flask, add enough lead subacetate to clarify; throw down the excess of lead with sodium sulphate solution; render slightly acid with a few drops of dilute acetic acid. Then dilute to 100 cc, filter, and polarize in a 200 mm tube. Reading represents (P) in the formula.

By means of a pipette, transfer 50 cc of the filtrate to a 100 cc flask, add 20 cc distilled water, and 5 cc concentrated hydrochloric acid. (Should contain 38.8 per cent of HCl.) Mix by a circular movement of the flask. Place a thermometer in the flask, bulb of thermometer near the center of the bulb of the flask. Put the flask in a water bath and regulate the temperature so that the solution will reach 67° to 69° C. in five minutes. Maintain this temperature for exactly five minutes longer, keeping the heat as near 69° as possible. Remove the flask, cool contents quickly to 17.5°, and make up to 100 cc with water. Filter if necessary. Avoid the use of bone black if possible. Polarize the solution in a 200 mm "inversion" tube. Use an accurate thermometer. Make the invert reading at exactly 20° C. Invert reading multiplied by 2 equals (I) in the formula.

Calculate results according to Wortman's formulæ:

$$S = \frac{0.9598\ P - 1.85\ I - 0.277\ N}{1.5648}$$

$$R = \frac{P - S + 0.3103\ N}{1.85}$$

In which—

P is polarization of normal solution before inversion.
I is invert reading multiplied by 2.
N is per cent reducing sugars.
S is per cent sucrose.
R is per cent raffinose.

The chemists of four experiment stations reported results. I am indebted to Prof. Elton Fulmer and to his assistant, Mr. W. H. Heileman, for a very complete report; to Messrs. Blouin, Winter, Kedzie, Miller, Munson, Chicquelin, Heuck, and Egge for their cooperation in this work, and to Prof. H. H. Nicholson and Dr. Wiley for valuable suggestions.

The results of the experimental work carried on this year, together with such deductions as the referee has been able to make, follow:

DETERMINATION OF WATER AND TOTAL SOLIDS.

The question of the best method of determining percentage of water in molasses and sugars is still a mooted one. It is certainly the most vexatious problem with which the sugar chemist has to deal. This association has considered the matter for a number of years, but as yet has come to no decisive conclusions.

Only two methods of desiccation were studied this year. One of these methods, the "Carr and Sanborn," has been adopted provisionally, but neither of them has received much attention from the association's chemists. The method of drying with quartz sand is one that is commonly employed in sugar-house laboratories, and has been found to yield good results so far as comparative work is concerned. The molasses experimented with this year contained such a high percentage of mineral matter and organic non-sugars that it was considered useless to try the method of dilution and "spindling."

The following results were reported:

Percentage of water by the Carr and Sanborn method.

Analyst.	Weight of dry matter obtained.	Water.	Average.
	Grams.	*Per cent.*	*Per cent.*
R. E. Blouin, La. Sugar Exp. Sta	1.0908	23.88	23.66
	1.1534	23.45	
Geo. Chicquelin, La. Sugar Exp. Sta	0.9990	23.57	23.45
	1.0300	23.34	
W. H. Heileman, Wash. Exp. Sta	1.8313	24.12	24.15
	1.7672	24.05	
	1.9438	24.04	
	2.1800	24.01	
	2.2139	24.15	
	2.3841	24.17	
	2.2712	24.25	
	2.5299	24.29	
	2.6535	24.19	
R. S. Hiltner, Nebr. Exp. Sta	1.3274	24.28	24.33
	1.3818	24.53	
	1.1811	24.31	
	1.0665	24.23	
H. K. Miller, N. C. Exp. Sta	.....	24.80	24.88
	.....	24.96	
L. S. Munson, Mich. Exp. Sta	1.5086	24.02	23.71
	2.2227	23.40	

Percentage of water.

[Desiccation with quartz sand.]

Analyst.	Weight of dry matter obtained.	Water.	Average.
	Grams.	*Per cent.*	*Per cent.*
R. E. Blouin, La. Sugar Exp. Sta	2.2950	23.11	23.12
	2.2903	23.13	
Geo. Chicquelin, La. Sugar Exp. Sta	2.3840	23.38	23.32
	2.8700	23.27	
O. H. Egge, Nebr. Exp. Sta	1.9004	24.40	24.30
	2.9766	24.21	
W. H. Heileman, Wash. Exp. Sta	1.5229	24.23	24.23
	0.7916	24.37	
	2.3570	24.18	
	2.3258	24.14	
R. S. Hiltner, Nebr. Exp. Sta	3.1303	24.15	24.02
	3.1325	24.02	
	3.0947	24.13	
	3.3901	23.90	
	3.9714	24.03	
	3.8068	23.93	
H. K. Miller, N. C. Exp. Sta	.....	23.97	24.07
	.....	24.18	
L. S. Munson, Mich. Exp. Sta	2.9793	23.01	23.10
	2.9034	23.18	

Taken as a whole, these results are certainly encouraging, but unfortunately the number of determinations made is so small that no definite conclusions can be drawn.

By reference to the above tables, it may be seen that 14 of the 21 of the percentages, as given by the Carr and Sanborn method, are included between 24.53 per cent and 24.02 per cent, a difference of 0.51 per cent, or an average of 24.16 per cent. By the quartz-sand method, 14 of 22 results given lie between 24.40 per cent and 23.90 per cent, a difference of 0.50 per cent, or an average of 24.15 per cent. It may be safely concluded from this that if we desire simply a comparative method either one of these schemes may be used.

The time required for desiccation seems to bear no direct relation to the amount of material taken. It seems to depend rather upon the kind of apparatus used (ovens and capsules), to the proportion of pumice or sand used, and to details of manipulation. This is indicated in the following table:

Time required for desiccation.

Analyst.	Method.	Weight of sample.	Hours required for drying.	Ratio.
		Grams.		
W. H. Heileman, Wash. Exp. Sta	Carr and Sanborn	2.5420	10	1:3.9
		3.1806	12	1:3.8
L. S. Munson, Mich. Exp. Sta	do	1.9856	23	1:11.5
		2.9094	30	1:10.0
R. S. Hiltner, Nebr. Exp. Sta	do	1.5496	10	1:6.4
		2.1992	14	1:6.3
W. H. Heileman, Wash. Exp. Sta	Quartz sand	2.0100	11	1:5.8
		1.0467	11	1:10.5
		3.0873	17	1:5.5
L. S. Munson, Mich. Exp. Sta	do	3.8247	36	1:9.4
R. S. Hiltner, Nebr. Exp. Sta	do	5.1160	24	1:4.4
		4.1460	25	1:6.0

ASH DETERMINATIONS.

All of the official methods for ash determinations, except the method for soluble ash, were studied this year. The results are as follows:

METHOD A.—*Ignition with sulphuric acid.*

Analyst.	Weight of sample.	Ash (sulphate).	Average.	Ash ($\frac{1}{10}$ correction).	Average.
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
R. E. Blouin, La. Sugar Exp. Sta	5.2605	8.58	8.56	7.72	7.70
	3.8385	8.54		7.69	
Geo. Chicquelin, La. Sugar Exp. Sta	3.7865	8.54	8.53	7.69	7.68
	3.3000	8.53		7.68	
W. H. Heileman, Wash. Exp. Sta	.....	8.30	8.04	7.47	7.24
	.....	7.83		7.05	
	.....	7.94		7.15	
	.....	8.02		7.22	
	.....	8.10		7.29	
R. S. Hiltner, Nebr. Exp. Sta	5.2371	8.23	8.48	7.41	7.64
	5.2973	8.62		7.76	
	6.5056	8.44		7.60	
	5.2898	8.65		7.79	
C. H. Heuck, Nebr. Exp. Sta	3.4691	8.66	8.60	7.80	7.74
	4.5140	8.64		7.78	
	3.2983	8.45		7.61	
	4.4246	8.66		7.80	
H. K. Miller, N. C. Exp. Sta	.....	8.74	8.74	7.87	7.87
	.....	8.74		7.87	
L. S. Munson, Mich. Exp. Sta	.....	5.45	[1] 5.45	4.91	[1] 4.91
General averages	.....	.....	8.49	.....	7.64

[1] Not included in the averages.

METHOD B.—*Alberti-Hempel.*

Analyst.	Weight of sample.	Ash.	Average.
	Grams.	*Per cent.*	*Per cent.*
R. E. Blouin, La. Sugar Exp. Sta	3.2795	5.32	5.24
	3.4431	5.16	
Geo. Chicquelin, La. Sugar Exp. Sta	3.1950	5.27	5.21
	2.9382	5.16	
W. H. Heileman, Wash. Exp. Sta	.....	5.15	5.18
	.....	4.76	
	.....	5.09	
	.....	5.09	
	.....	5.43	
	.....	5.37	
	.....	5.35	
	.....	5.25	
C. H. Heuck, Nebr. Exp. Sta	4.8957	4.96	5.16
	4.8949	5.37	
R. S. Hiltner, Nebr. Exp. Sta	4.1272	5.19	5.17
	4.1227	4.94	
	4.0791	5.20	
	4.4552	5.14	
	5.2275	5.28	
	5.0046	5.30	
H. K. Miller, N. C. Exp. Sta	.....	5.69	[1]5.52
	.....	5.20	
	.....	5.50	
	.....	5.72	
L. S. Munson, Mich. Exp. Sta	.....	5.15	5.15

[1] Omitted from average.

METHODS C AND D.—*Direct ignition.*

Analyst.	Ash. (Incineration without oil).	Average.	Ash. (Incineration with oil).	Average.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Geo. Chicquelin, La. Sugar Exp. Sta	7.05	7.05	.....	.....
	7.05		.....	
W. H. Heileman, Wash. Exp. Sta	6.93	6.97	7.05	7.10
	6.97		7.15	
	7.03		.....	
	6.91		.....	
	6.99		.....	
R. S. Hiltner, Nebr. Exp. Sta	6.92	6.89	6.72	6.92
	6.57		7.05	
	6.93		7.01	
	7.15		.....	
H. K. Miller, N. C. Exp. Sta	7.18	7.08	.....	.....
	6.98		.....	
L. S. Munson, Mich. Exp. Sta	6.54	6.54	.....	.....

METHOD E.—*Ignition with zinc oxid.*

Analyst.	Ash.	Average.
	Per cent.	*Per cent.*
W. H. Heileman, Wash. Exp. Sta	7.49 7.27	7.38
R. S. Hiltner, Nebr. Exp. Sta	6.68 6.71	6.69
General average		7.03

METHOD F.—*Ignition with benzoic acid.*

Analyst.	Ash.	Average.
	Per cent.	*Per cent.*
W. H. Heileman, Wash. Exp. Sta	6.90 6.88 [1]6.65 6.78	6.85
R. S. Hiltner, Nebr. Exp. Sta	6.80 7.14 [1]6.31 [1]6.54	6.97
General average		6.91

[1] Ash fused; not included in average.

Summary of results.

Method.	Average per cent of ash.
Ignition with sulphuric acid	7.64
Ignition with quartz sand	5.18
Direct ignition	6.90
Direct ignition (oil added)	7.01
Ignition with zinc oxid	7.03
Ignition with benzoic acid	6.91

By the same methods the results of these determinations of ash in beet molasses compare in a general way with the results of analyses of cane products which were carried on a few years ago by the association's chemists. (See Bulletin 38, p. 150.) This would indicate that for ash no special method nor modifications of present methods are necessary for beet products.

The percentage of "sulphated" ash is, as usual, too high as compared with that obtained by other methods, signifying that the one-tenth correction proposed by Scheibler is too small. In spite of this fact I take it that the association would still be unwilling to drop this process from the list of official methods or to consider a change of factor. This method may always be relied upon to yield concordant results. Incineration in the presence of sulphuric acid is readily effected and the resulting ash is but little changed by high temperatures. Moreover, "sulphated" ash is usually employed in calculating rendement, another argument in favor of retaining the method.

The most uniform results were obtained by ignition with sand. As may be seen from the above table, this method yields a much lower percentage of ash than any other of the methods tried. The reason for this is understood to be that the organic acids are replaced by silicic acids, leaving little or no carbonates; the salts of the inorganic acids, with the exception of nitrates, remain unchanged.

The results obtained by the direct ignition of the molasses and by ignition with benzoic acid agreed more closely than was anticipated. The difficulty usually experienced is in controlling the temperature of the muffle to prevent loss of salts by volatilization. By a few experiments the referee has attempted to show the effect of high temperatures upon the ash of the molasses in question.

Temperature.	Sulphate method.	Alberti-Hempel method.	Direct ignition.	Ignition with oil.	Ignition with benzoic acid.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Very low red heat till completely ashed	7.76	5.20	6.93	6.72	6.80
Bright red heat for fifteen minutes	7.75	5.06	6.73	6.38	6.36

The addition of a small amount of olive oil to the sample before incineration is a convenience, simply preventing excessive foaming. As to the value of the methods of ashing with benzoic acid and with zinc oxid no conclusions can be drawn from the present series of experiments.

REDUCING SUGARS.

In commercial and factory control the amount of reducing sugars in beet products is regarded as nil and is almost never determined. The results obtained, however, show the presence of these sugars in small quantities and also show the value of the gravimetric methods, so called, for the exact determination of them. In fact the volumetric methods, such as Soxhlet's modified method, may not be used, owing to the large proportion of sucrose.

The process of estimating the amount of reduced copper by titrating with permanganate solution is simple and rapid and yields results that agree closely with those by the electrolytic method.

The results obtained by almost all of the analysts by both methods agree very closely. These results are presented in the following table:

METHOD A.—*Reduced copper determined by electrolysis.*

Analyst.	Weight of sample used.	Weight of copper.	Reducing sugars calculated as dextrose.	Average.
	Grams.	*Gram.*	*Per cent.*	*Per cent.*
R. E. Blouin, La. Sugar Exp. Sta	10	0.0380	0.199	0.199
W. H. Heileman, Wash. Exp. Sta	10	.0405	.211	.196
	10	.0330	.175	
	10	.0375	.196	
	10	.0345	.183	
	10	.0425	.221	
C. H. Heuck, Nebr. Exp. Sta	10	.0536	.277	.277
R. S. Hiltner, Nebr. Exp. Sta	6.500	.0371	.298	.298
H. K. Miller, N. C. Exp. Sta	.....	.....	.120	.120
L. S. Munson, Mich. Exp. Sta	15.7544	.062	.202	.199
	17.1460	.0654	.196	

METHOD B.—*Reduced copper determined by titration with permanganate solution.*

Analyst.	Weight of sample used.	Copper equivalent.	Reducing sugar.	Average.
	Grams.	*Gram.*	*Per cent.*	*Per cent.*
W. H. Heileman, Wash. Exp. Sta.	10	0.0383	0.200	0.196
	10	.0379	.199	
	10	.0375	.196	
	10	.0363	.190	
R. S. Hiltner, Nebr. Exp. Sta.	7.9962	.0390	.300	.305
	16.1967	.0990	.311	

DETERMINATIONS OF SUCROSE AND RAFFINOSE.

There certainly can be no doubt as to the presence of raffinose in some of the by-products of American beet-sugar houses. (See Journ. of Am. Chem. Soc., 1897.) Moreover, there can be no doubt as to the presence of small amounts of reducing sugars in low-grade factory products, even when obtained under normal conditions.

It is common practice in cane-sugar houses to determine sucrose by double polarization, using for calculations Clerget's formula. This gives correct values when sucrose and invert sugar are alone present. When, however, sucrose is associated with raffinose and when, as is commonly the case with beet products, invert sugar is absent, the following formulæ proposed by Creydt are employed:

$$S = \frac{0.5188\,P - I}{0.845} \qquad R = \frac{P - S}{1.85}$$

Beet-sugar chemists, both in this country and in Europe, usually employ Clerget's formula when raffinose is absent, and the Creydt formula when this sugar is present. In each case the presence and influence of any reducing sugar is ignored.

The referee's purpose in asking for the estimation of sucrose and raffinose in the samples distributed was to determine the practicability of existing methods when applied to such material, and also to find out if possible the effect of a small amount of reducing sugars upon the double polarization. The extent of error, if any, that results from disregarding the influence of the reducing sugars should appear by calculating the percentages of sucrose and raffinose by Wortman's and by Creydt's formulæ. The results as reported follow:

Analyst.	Normal polarization before inversion.	Normal polarization after inversion.	Reducing sugar (dextrose).	Sucrose (Wortman).	Raffinose (Wortman).	Sucrose (Creydt).	Raffinose (Creydt).
			Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
R. E. Blouin	57.35	14.6	0.199	52.40	2.70	52.48	2.63
Geo. Chicquelin	57.1	14.4	.215	52.02	2.78	52.09	2.70
W. H. Heileman	56 58	16.1	.196	53.70	1.59	53.79	1.50
C. H. Heuck	57.0	13.6	.277	51.64	2.94	51.09	3.19
R. S. Hiltner	56.9	14.0	.300	51.30	3.07	51.50	2.91
H. K. Miller	.....	.....	.120	49.50	4.40	.....	.....
L. S. Munson	56.0	12.4	.199	48.97	3.84	49.05	3.75

The exact significance of such a variety of results is not clear. On the whole, the direct polariscopic readings as reported agree fairly well, but the differences in polarization after inversion are large and at the same time difficult to explain.

The molasses in question was an extremely difficult substance to clarify, making optical determinations of the sugars not at all easy. This fact would doubtless justify a certain slight variation in polarimetric tests, but such a wide variety of polariscopic readings as given above can not be explained nor justified in this way. The referee's observations in experimenting with this molasses were that a wide range of results could be obtained by changing slightly the time and temperature conditions of the method of inversion, but by following out one particular scheme in detail concordant results would in each case follow. Other analysts have probably experienced the same thing, a fact which points to the necessity of further investigation of this subject.

Besides studying details of a method for double polarization, it would be well for the association to consider what formulæ for calculation shall be used for various sugar-house products. One thing is certain, we may not properly apply the same formulæ to both cane and beet products. The presence of raffinose in the latter often precludes the use of Clerget's formula. It would be rash to decide upon these points from the limited data herein reported. We must as an association either investigate the question for ourselves or else decide from the experiments of individual investigators. From the results given in the last table, it would seem unnecessary to employ Wortman's formula for calculating sucrose and raffinose, the percentage of reducing sugars being too small to be of any serious consequence.

The referee would ask that the following recommendations be considered:

(1) That the Carr & Sanborn method for determination of water in molasses, etc., be adopted as an official method.

(2) That the method of desiccation with quartz sand be made an optional or a provisional method, and that the association experiment further with it.

(3) That to the method for determining per cent of water from the density a note be added to the effect that this method does not apply to low grade sugar products.

(4) M. Lucien, in describing his method for ash by incineration with zinc oxid, proposes to use 0.050 gram zinc oxid with 5 grams sugar or with 3 grams molasses. (Bul. de l'Assoc. des Chem., vol. 6, p. 356.) On the other hand, our official method calls for the same amount of zinc oxid, 0.050 grams, with 50 grams sugar or 25 grams molasses. I do not understand the reason for this discrepancy. My own experience has been that the proportion of zinc oxid, as suggested in our official method, is entirely too small. It is probable that the difference is due to a typographical error in our method, and if so, it should, of course, be corrected. If we divide by 10 the weight of sample to be employed, we will approximate the proprotions of sugar to zinc oxid proposed by Lucien.

(5) That methods (b) and (c) for determination of copper in the cuprous oxid obtained in determining reducing sugars be dropped from the list of official methods. This will leave us one electrolytic method, the Formanek, which is conceded to be the simplest and most rapid of the three.

(6) That in the text of method (e) for determination of reduced copper, directions be inserted for preparing the permanganate solution used. The number of grams of potassium permanganate required per liter should be stated. (Sol.=4.976 3 grams per liter.)

(7) That Creydt's method (Zeit Rübenzack, Ind. 38, 867) for determination of sucrose and raffinose be adopted provisionally and added to the list of "methods by inversion."

In the discussion of this paper Mr. Bigelow called attention to the fact that in the volumetric method depending upon the use of a permanganate solution and ferrous salt, the permanganate should be carefully compared with the ferrous sulphate.

After further discussion of the paper, the president recognized Mr. Jaffa, for the purpose of extending his invitation to the association to hold its next meeting in San Francisco. Mr. Jaffa assured the association of the hospitable welcome which it would receive upon the Pacific coast, and spoke of the advantages of meeting now and then in other localities than Washington. The members would find upon the Pacific coast conditions prevailing which are entirely different from those upon the Atlantic side of our country, and they would not only have great pleasure but also profit from personal inspection of the methods of work in vogue beyond the Rocky Mountains. Among other things, he said the members would be delighted to meet Professor Hilgard, the state of whose health prevented him from attending the meetings at such a distance.

Responding to questions in regard to the rates, Mr. Jaffa said that while he could not say what they would be he could say they would be about the smallest that had ever been inaugurated. A great many associations had already decided to meet on the Pacific coast during the summer and autumn of 1899, and the rates which could be had for the large associations would apply also to the small ones.

The association was much impressed with Mr. Jaffa's presentation of the advantages which would come to it by holding a session on the Pacific, and it was voted that the matter be left to the executive committee, with power to act.

DELEGATES TO NATIONAL PURE FOOD AND DRUG CONGRESS.

At the conclusion of Mr. Jaffa's address the secretary called attention to a communication from Mr. A. J. Wedderburn, corresponding secretary of the National Pure Food and Drug Congress, stating that the second annual session of this congress would convene in Washington on the 18th of January, and the association was requested to name five delegates and five alternates to represent it in the congress. Attention was also called to the fact that each official agricultural chemist of the country, by virtue of his office, was entitled to membership in the congress and was heartily invited to be present.

The president appointed the following delegates: H. W. Wiley, A. L. Winton, W. D. Bigelow, H. J. Wheeler, L. L. Van Slyke, and E. A. de Schweinitz. Alternates: W. O. Atwater, A. B. Peebles, H. B. McDonnell, H. E. Alvord, H. J. Patterson, and R. A. Emerson.

On the suggestion of Mr. Wheeler it was voted that the secretary of the association be requested to rewrite the methods of analysis preparatory to having them reprinted, with the object of condensing them so far as would be possible without sacrificing clearness. It was also voted that he be allowed to select as many assistants in the conduct of this work as might be necessary to secure its prompt completion.

It was suggested by Mr. C. B. Coates that the agricultural colleges and experiment stations should pay the expenses of the delegates to attend the meetings of the association. This opinion was heartily indorsed by other members of the association, with expressions of a belief that the portion of the money used for experimental work could not be better applied than by sending the workers to these conventions, where they would meet those engaged in similar work, compare notes, make acquaintances, and, in general, fit themselves all the better for the proper conduct of their work.

At 3.15 it was voted to adjourn, as it seemed to be impossible, even with the strict attention to business which had been indulged in, to finish the business of the association before Monday.

THIRD DAY.

MONDAY—MORNING SESSION

The association reconvened at half-past nine.

The president congratulated the members on having present the honorable the Secretary of Agriculture. A few years ago, many might remember, when the experiment stations were in their infancy, the meetings of this association were not regarded with any favor and they were not patronized by the distinguished gentlemen at the head of affairs. This state of things has largely passed away, and now our association is honored annually by the presence of a Cabinet officer for the purpose of expressing his interest in our work and giving us words of encouragement. The presence of the Secretary of Agriculture is an evidence that the spirit of research and discovery is triumphant throughout the whole area where scientific agriculture is practiced and promoted by the United States Government. A part of the power which the experiment stations have acquired is shown in the fact that the President of the United States selected the director of an agricultural experiment station for the responsible position of Secretary of Agriculture. This is a matter of which we should all be proud, and for this especial reason, as well as for many others, we are glad to have with us this morning the Secretary of Agriculture.

The Secretary then addressed the Association.

REMARKS OF SECRETARY WILSON.

The Secretary commenced his address by recalling the visit of Dr. Armstrong, a distinguished English chemist, who came to Washington expecting to spend a half day in looking over the scientific divisions of the Department of Agriculture. After having been here a week he expressed the belief that he could not even begin to comprehend the great scientific work which the Department was doing if he should stay a month.

The Secretary, continuing, said:

I lately asked Dr. Wiley for the reason why we imported so many chemicals. I noted down some of the points which he mentioned and will refer to them further on.

Very little progress could be made in any vocation of life without the help of the chemist. He is indispensable to the farmer. The composition of soils and their supply of plant food are the first lesson the producer should learn. Where fertilization is necessary and the market must be sought, the chemist is the only protector the purchaser can rely on. The feeder nowadays finds it necessary to know the composition of every nutrient, adapt it to the age of the animal and the purpose he

has in view. The ration that will fatten the steer will put the cow dry, or what is suitable for the yearling is wasted on the mature animal. Plants vary in protein, from young clover to old timothy. The growing colt needs the composition of the former in his ration, the idle horse does well on the latter. Between the nutritive ratios of the two plants is latitude sufficient to waste the profits of labor. Farming is not profitable until we learn to feed, and our first teacher is the chemist.

We should know how to feed ourselves and others. People tell us of the sufferings of those who have only army hard-tack to eat, not having learned that, after milk, it comes nearest being complete food. Southern workmen had corn bread, fat pork, and molasses until the chemist told them to grow beans, keep cows, and balance their rations.

The markets abound with mill feeds that vary in composition as much as oil meal and wheat straw. We look to the report of the chemist to direct us in buying. It is settled that we must teach chemistry to mothers and cooks, so that they will know how to vary the rations for a child, a youth, a laboring man, and an octogenarian.

The chemist has analyzed sugar beets for us grown in most of the States, and tells us where conditions are suitable for the investment of $200,000,000 of capital, now lying idle or being loaned in Europe, to build factories where 1,800,000 tons of sugar may be made and save $100,000,000 to the United States and give good wages to hundreds of thousands of workmen.

We paid $41,470,711 for chemicals, drugs, and dyes the last fiscal year; five-eighths of these came in free; three-eighths paid duty. Somebody blundered. We have educated chemists, capital and raw material in great abundance, and should provide these things for ourselves, as we make steel and tin. An extensive island area is being added to our domain, where the scientist has made little research. The chemist should go with the botanist, the pathologist, and the roadmaker. We must learn of the soils, their origin and composition, before we can tell what they can permanently and profitably produce.

The Secretary's address was greeted with hearty applause.

At its conclusion the president called for the report of the referee on tannin. The referee, Mr. J. H. Yocum, read the following report:

REPORT ON TANNINS.

J. H. Yocum, *Referee.*

The comparative work on tannins during the past year is of considerable interest, inasmuch as there are no less than twenty individuals contributing, and in addition to the results obtained on the samples sent out, Messrs. Carr, Forbes, Eachus, and Alsop present some interesting data relative to the method, which are embodied in this report.

Two lots of samples were sent out, the first at the beginning of the year being a commercial chestnut-wood extract and a hemlock liquor, and with these were sent two makes of hide powder, one from Vienna and the other from Ridgway, Pa.

It was requested that the official method be followed, and that if other than the official method were in use by the collaborator results by it be also sent in. The hemlock liquor was thoroughly filtered before being sent out, and showed no precipitate at 40° F. and contained no reds at the time of shipment.

The second lot of samples was an extract made by the referee from grape sugar and gallotannin and contained no precipitate when shipped, but upon standing some three months did precipitate some insoluble material. The reports, however, were all in before this was noticed, and it is not probable that there was any insoluble material in this extract when the various analyses were made. Vienna hide powder was sent with this sample.

The results obtained are as follows:

Chestnut-wood extract.

Name.	Hide.[1]	Moisture.	Total solids.	Soluble solids.	Non-tannins.	Tannins.	Reds.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Pr ct.*
Allaire	A	59.59	40.41	38.45	14.05	24.40	1.96
	B	.....	.....	.....	14.34	24.11	
Alsop	A	59.17	40.56	37.99	13.75	24.24	2.57
	B	.....	.....	.....	13.69	24.30	
Carr	A	59.49	40.53	37.66	15.53	22.13	2.87
	B	.....	.....	.....	15.72	21.94	
Eachus	A	59.17	41.06	39.14	14.21	24.93	1.92
	B	.....	.....	.....	14.36	24.78	
Eitner (Weiss)	A	58.16	41.84	40.89	14.45	26.14	.95
	B	.....	.....	.....	14.55	26.34	
Forbes	A	58.22	41.48	40.12	13.92	26.20	1.36
	B	.....	.....	40.09	13.86	26.23	1.39
Haley	A	58.96	41.04	38.62	13.88	24.74	2.42
	B	.....	.....	.....	14.06	24.56	
Hurty	A	58.11	41.89	39.38	14.87	24.51	2.51
	B	.....	.....	.....	14.80	24.58	
Krug	A	59.60	41.05	39.54	14.16	25.38	1.51
	B	.....	.....	.....	14.78	24.76	
Lowell	A	58.15	41.85	39.50	14.65	24.85	2.35
	B	.....	.....	.....	14.75	24.75	
McDowell	A	58.95	40.87	38.70	14.03	24.67	2.17
	B	.....	.....	.....	14.64	24.06	
Proctor	A	58.97	41.03	38.10	15.55	22.55	2.93
	B	.....	.....	.....	15.30	22.80	
Small	A	59.21	40.72	38.22	14.57	23.65	2.50
	B	.....	.....	.....	14.22	24.00	
Tiffany	A	58.89	41.11	38.97	13.46	25.51	2.14
	B	.....	.....	.....	13.69	25.28	
Westenfelder[2]	.....	59.05	41.44	38.96	14.33	24.63	2.99
Parker[2]	A	.....	.....	40.10	12.20	27.90	
	B	.....	.....	.....	14.30	25.80	
Touse[2]	A	.....	.....	40.16	12.25	27.91	
	B	.....	.....	.....	14.50	25.66	
Yocum	A	59.20	40.59	38.20	13.61	24.59	2.39
	B	.....	.....	.....	13.92	24.28	
Mean	A	58.89	41.07	38 98	14.31	24.67	2.09
Maximum	A	59.60	41.89	40.89	15.55	26.44	2.93
Minimum	A	58.11	40.41	37.66	13.46	22.13	.95
Mean	B	.....	.....	.....	14.52	24.46	
Maximum	B	.....	.....	.....	15.72	26.34	
Minimun	B	.....	.....	.....	13.69	21.94	
Maximum difference from minimum	A	1.49	1.48	3.23	2.09	4.31	1.98
	B	.....	.....	.....	2.03	4.40	
Maximum difference from mean	A	.71	.82	1.91	1.24	2.54	1.14
	B	.....	.....	.....	1.20	2.52	

[1] Hide powder "A" was the Vienna product; hide powder "B" was the Ridgway product.
[2] Not having followed official method, are not included in average.

Hemlock liquor.

Name.	Hide.[1]	Total solids.	Soluble solids.	Non-tannins.	Tannins.	Reds.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per ct.*
Allaire	A	4.57	4.44	2.02	2.42	0.13
	B	.....	.....	2.10	2.34	.....
Alsop	A	4.72	4.57	2.20	2.36	.15
	B	.....	.....	2.12	2.45	.....
Carr	A	4.63	[2]4.63	2.32	2.31	.....
	B	.....	.....	2.35	2.28	.....
Eachus	A	4.78	4.68	2.13	2.55	.10
	B	.....	.....	2.22	2.44	.....
Eitner (Weiss)	A	4.77	4.63	2.16	2.47	.14
	B	.....	.....	2.18	2.45	.....
Forbes	A	4.69	4.67	2.14	2.53	.02
	B	.....	.....	2.18	2.49	.....
Haley	A	4.83	4.63	2.35	2.28	.20
	B	.....	.....	2.17	2.46	.....
Krug	A	4.54	4.48	2.16	2.32	.06
	B	.....	.....	2.13	2.35	.....
Lowell	A	4.84	4.74	2.20	2.54	.10
	B	.....	.....	2.24	2.50	.....
McDowell	A	4.74	4.63	2.18	2.45	.11
	B	.....	.....	2.23	2.40	.....
Proctor	A	4.72	4.53	2.25	2.28	.19
	B	.....	.....	2.24	2.29	.....
Small	A	4.64	4.55	2.16	2.39	.09
	B	.....	.....	2.17	2.38	.....
Yocum	A	4.71	4.66	2.14	2.52	.05
	B	.....	.....	2.13	2.53	.....
Parker	A	4.80	[2]4.80	2.00	2.80	.....
	B	.....	.....	2.20	2.60	.....
Mean	A	4.71	4.60	2.17	2.43	.11
Maximum	A	4.84	4.74	2.35	2.55	.19
Minimum	A	4.54	4.44	2.00	2.28	.02
Mean	B	.....	.....	2.19	2.41	.....
Maximum	B	.....	.....	2.35	2.53	.....
Minimum	B	.....	.....	2.10	2.28	.....
Maximum difference from mean	A	.17	.16	.18	.15	.09
	B	.....	.....	.16	.13	.....

[1] Hide powder "A" was the Vienna product; hide powder "B" was the Ridgway product.
[2] Not filtered for soluble solids and hence not included in averages of soluble solids and tannins.

Theoretical extract.

Name.	Moisture.	Total solids.	Soluble solids.	Non-tannins.	Tannins.	Reds.	Red tannin.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per ct.*	*Per cent.*
Alsop	61.20	38.82	37.32	19.18	18.14	1.50	19.64
Carr	61.97	37.91	[1]37.91	18.66	19.25	.....	19.25
Eachus	61.60	38.43	37.42	18.90	18.52	1.01	19.53
Forbes	60.57	39.43	[1]39.43	19.04	20.39	.....	20.39
Haley	60.69	39.23	37.46	18.64	18.82	1.77	20.59
Hurty	60.73	39.27	38.33	18.89	19.44	.94	20.38
Kerr	60.01	39.99	38.33	21.41	16.92	1.66	18.58

[1] Not filtered for soluble solids, hence not included in averages of soluble solids or tannins.

NOTE.—Hide powder was the Vienna product.

Theoretical extract—Continued.

Name.	Moisture.	Total solids.	Soluble solids.	Non-tannins.	Tannins.	Reds.	Red tannin.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per ct.*	*Per cent.*
McDowell	60.94	38.72	37.50	19.12	18.38	1.22	19.60
Nichols	61.35	38.65	36.88	18.68	18.20	1.77	19.97
Small	61.99	38.09	36.95	17.50	19.45	1.14	20.59
Yocum	61.05	38.20	36.80	17.90	18.90	1.40	20.30
Mean	61.10	38.79	37.44	18.90	18.54	1.35	19.89
Maximum	61.99	39.99	38.33	21.41	20.39	1.77	20.59
Minimum	60.01	37.91	36.80	17.50	16.92	.94	18.58
Maximum difference from minimum	1.98	2.08	1.53	3.91	3.47	.83	2.01
Maximum difference from minimum	1.09	1.20	.89	2.51	1.85	.42	1.31
Theoretical content of sample	62.23	37.77	37.77	17.58	20.19	.....	20.19

Mr. Pierre Allaire states that the official method was followed; that he considers hide powder "A" a little better than "B," and that he did not obtain a perfectly clear non-tannin filtrate from the extract. Mr. B. Weiss made the analysis of the sample sent to Mr. Eitner of the Vienna Research College for the Leather Industries. He states that he pursued the official method, and also reports his results from the filter-tube method. He obtained 13.90 per cent non-tannins on the chestnut-wood extract and 2.13 per cent non-tannins on the hemlock liquor by following the filter-tube method.

Dr. Hurty obtained 41 per cent total solids, 39.68 per cent soluble solids, 15.50 per cent non-tannins, and 24 18 per cent tannins by a method he published sometime ago in the Leather Manufacturer.

W. C. Tiffany: "Our method varies from the official in that we employ 10 instead of 15 grams dry hide powder, and we shake three times, using one-third of the hide powder each time." He reports from this method 41.11 per cent total solids and soluble solids—non-tannins from "A" hide powder, 12.32; "B," 12.90—giving respectively, 28.79 per cent and 28.21 per cent tannins.

Mr. Kerr states that the sample of grape sugar and gallotannin was clear and light brown in color when received, but before being analyzed was covered with a luxuriant growth of mold. He doubts the value of his results in consequence.

M. S. McDowell: "The extract (grape sugar and gallotannin) when dissolved made a clear solution, but upon filtering through S. & S. paper 1.22 per cent were removed. Am of the opinion that in all cases there is an absorption, greater or less, by the cellulose of the filter paper."

Mr. Lowell reports on the chestnut-wood extract 15 per cent non-tannins and on the hemlock liquor 2.23 per cent non-tannins by following the official method and using the kind of hide powder employed in his laboratory, which contained 0.0070 gram of material soluble in water, as per directions for obtaining same.

Mr. Kerr states that by using his laboratory hide powder, which contained 0.0080 gram soluble material, he obtained 21.41 per cent non-tannins from the grape-sugar extract.

Dr. J. Gordon Parker reports results obtained by Herolds Institute method: tannins 28.90 per cent, non-tannins 12.10 per cent, solids 40.10 per cent on the chestnut-wood extract; and tannins 2.80 per cent, non-tannins 2 per cent, solids 4.80 per cent on the hemlock liquor. He states: "You will notice that they (the analyses he reports) are all done by the hide-filter method. * * * None of the hide powders has been washed, as you instructed, an ordinary analysis being made according to the method laid down by the international association with Proctor's form of hide

filter, 5 grams of powder being used in each case. Mr. Touse, my colleague, employed the same process."

Dr. H. R. Proctor: "The method appears to be a good one, and the two hide powders give very concordant results; but I do not know that it is better than the 'International,' while the results are in every case lower. I do not regard this as a defect, though it is impossible with our present knowledge to say that either give the absolute percentage of tanning, or one more than the other."

By the "International" filter-tube method he obtains on the chestnut-wood extract: tanning matter 27.9 per cent, non-tannins 11.8 per cent, insoluble at 60° F. 1.9 per cent, water 58.4 per cent. By the Yorkshire College shake method: tanning matter, Vienna hide 24.1 per cent, Buffalo hide 22.4 per cent; non-tannins, Vienna hide 15.6 per cent, Buffalo hide 17.3 per cent. In this last method the powder is added in three successive portions, 6 grams powder used and 100 cc of liquor. The Buffalo powder is coarse-ground dry hide, heavy, granular, and unsuitable for the filter tube. In the filter method the first 30 cc passing through are thrown away, the next 50 cc evaporated for non-tannins. On the hemlock liquor, by the "International" method, Dr. Proctor obtained tanning matter 2.45 per cent, non-tannins 2.16 per cent; and by the Yorkshire method, tanning matter 2.25 per cent, non-tannins 2.35 per cent, results being identical from both Vienna and Buffalo hide.

Dr. Forbes reports non-tannins obtained by the use of Fiebing's hide powder in the chestnut-wood extract 13.15 per cent; in the hemlock liquor 2.05 per cent, and in the grape-sugar extract 19.05 per cent. All filtrations were done through No. 597 S. & S. paper, and all dryings at 100° C.

E. J. Haley finds 14.28 per cent non-tannins in the chestnut-wood extract from a hide powder of his own make. He reports the ash analysis of the two hide powders sent out:

	"A" hide.	"B" hide.
	Per cent.	*Per cent.*
Ash	0.19	0.36
Lime	.06	.05
Alumina	Trace.	Trace.
Ferric oxid	Trace.	Trace.

Mr. F. H. Small reports that his laboratory method does not vary from the official to any extent and hence does not report from it.

Mr B. D. Westenfelder: "I did not follow directions you sent out, but used my laboratory method. It is as follows: Dissolve in water at 40° C. in a 500 cc flask; place on water bath for fifteen minutes and agitate frequently; fill to mark with cold water; cool rapidly in cold water and make up to 1 liter; filter through 20 cm double-folded S. & S. No. 6298 filter paper; discard first 100 cc that passes through; refilter then till absolutely clear.

Wash hide powder with water containing acid sodium sulphite and sulphuric acid, then wash till absolutely free from soluble hide and saline matter, squeeze and shake. All residues dried in air oven at 104° to 106° C. for two hours." His results are:

	Chestnut-wood extract.	Hemlock liquor.	Grape-sugar extract.
	Per cent.	*Per cent.*	*Per cent.*
Solids	41.95	4.57	39.80
Soluble solids	38.96	4.57	39.80
Non-tannins:			
No. 1	13.07	2.13	19.53
No. 2	14.33	2.13	19.83

Non-tannin No. 1 wet hide powder was added, in two portions of 25 grams each; 50 grams wet hide total used. Non-tannin No. 2, 50 grams wet hide added at once, 200 cc of liquor used for shaking and 100 cc evaporated; wet hide powder contained 78 per cent moisture. In the case of the liquor only 40 grams of wet hide were used.

Mr. Oma Carr states that at 25° C. he obtained 2.09 per cent of reds in the chestnut-wood extract, and at 10° C. 3.66 per cent reds, an increase of 1.57 per cent. He also states that at 25° C. the hemlock liquor was clear, but at 10° C. he found 0.20 per cent of reds. By dissolving an extract according to the method (20 grams to the liter), and by dissolving it so that it will make a liquor of 1.02 sp. gr., he finds:

	Official.	At 1.02 sp. gr.	Difference.
	Per cent.	*Per cent.*	*Per cent.*
Total solids	40.63	40.43	0.10
Soluble solids	37.66	37.38	.28
Reds	2.87	3.05	.18
Tannin	22.03	21.71	.32
Non-tannins	15.63	15.66	.03

This liquor of 1.02 sp. gr. gave soluble solids, at 25° C. 37.38, and at 8° C. 35.94, a difference of 1.44. Mr. Carr presents the following data regarding the drying of the grape-sugar extract:

Dried in air at 100° C.

Solids (direct).			Solids (indirect).			Non-tannins.		Tannins.	
Time.	Interval.	Per cent.	Time.	Interval.	Per cent.	Time.	Interval.	Per cent.	Per cent.
2	.....	40.46	3	.....	39.26	.....	.....	.....	.....
4	2	40.00	.....	.....	.....	.....	.....	.....	.....
10	6	39.04	.....	.....	.....	.....	.....	.....	.....
12	2	38.99	.....	.....	.....	16	.....	19.67	.....
28	16	38.48	24	21	38.28	24	8	19.46	18.82
32	4	38.35	.....	.....	.....	.....	.....	.....	.....
50	18	38.03	42	18	37.91	45	21	18.66	19.25
56	6	37.93	50	12	37.81	64	19	18.10	19.71
96	40	37.51	90	40	37.22	112	48	17.22	20.00
136	40	37.29	138	48	36.99	136	24	16.92	20.07
Theoretical contents of sample..	.....	.....	.....	.....	37.77	.....	.....	17.58	20.19
Maximum decrease ..	3.17	.....	.....	2.27	.....	.....	2.75	.....	

"The non-tannins show a rate of decomposition about twice as great as the solids as a whole—precisely as it should be, considering that the non-tannins are largely left handed sugars.

"The absurdity of drying five days without constant weight is manifest. In my opinion, the water is wholly expelled from residues of 500 milligrams or less inside of four hours and any loss thereafter is chargeable to decomposition. These sugars of the dextrose order do not oxidize appreciably, while their rate of decomposition is wholly dependent upon the temperature used in their dehydration. It is possible to select from the above table percentages in fair accordance, but I hold that such arbitrary selection is absolutely without apology where the rate of decrease continues thereafter, whether more slowly or not.

"It seems to me wisely consistent with the best interest of the method to stipulate that extracts and other materials containing large quantities of invert sugars must be dried in vacuo at 70° C. The theoretical sample has served the exceedingly useful purpose of proving the inapplicability of ordinary dehydration methods to its desiccation. If the objection be made that chemists are not supplied with means of

vacuum drying, it is a wholly consistent thing to require them to provide it. In the analysis of sugar, ore, milk, etc., the means have been adapted to the method, and the polariscope, assay balance, and Babcock machine are essentials.

"When it comes thus to a choice between a method vitally defective and one in which special apparatus must be provided, which is necessary in the operation of a method which has the advantage of accuracy, there seems hardly room for debate."

Mr. Eachus presents the following data on drying:

"Twelve portions of 100 cc of well-mixed extract solution (20 grams to the 100 cc) were evaporated to dryness. Six of these were dried on the top shelf of a hot-air oven; the other six were dried on the bottom shelf of the same oven. At the end of three hours one dish from each shelf was weighed, and at one hour intervals thereafter a dish was weighed from each shelf. The temperature of the top shelf was 100° C, and of the bottom shelf 125° C.

"Twelve portions of 100 cc of non-tannins from the same solution were treated similarly.

Time.	Solids.			Non-tannins.		
	100° C., top shelf.	125° C., bottom shelf.	Difference.	100° C., top.	125° C., bottom.	Difference.
	Gram.	*Gram.*	*Gram.*	*Gram.*	*Gram.*	*Gram.*
After 3 hours	0.8399	0.8218	0.0181	0.3036	0.2843	0.0193
After 4 hours	.8275	.8152	.0123	.3052	.2842	.0210
After 5 hours	.8280	.8111	.0169	.3051	.2855	.0196
After 6 hours	.8247	.8099	.0148	.3044	.2842	.0202
After 7 hours	.8244	.8009	.0235	.3027	.2820	.0207
After 8 hours	.8283	.8027	.0256	.3072	.2863	.0209
Average			.0185			.0203

"From this it is seen that variations are due to drying at various temperatures and that a constant weight is obtained by drying at 100° C. for from four to eight hours, but that a loss of non-tannins occurs by drying at 125° C. both in the total solids and non-tannin determinations, probably due to decomposition of the non-tannins. Variations in temperature within these limits affect the non-tannins largely without any considerable action upon the tannins. Variations then in drying tannin solutions are a function of the temperature, although no doubt long-continued drying at 100° C. would occasion oxidation of the tannin.

"Fourteen analyses of an extract were made and the drying done in various ways, always making it a point to dry the soluble solids and non-tannins under identical conditions and side by side in the drying oven.

Dried on—	Weight of soluble solids.	Weight of non-tannins.	Soluble solids.	Non-tannins.	Tannins.
	Gram.	*Gram.*	*Per cent.*	*Per cent.*	*Per cent.*
Top shelf hot-air oven, 4 hours at 100° C	0.8686	0.4426	43.43	26.12	17.31
Top shelf hot-air oven, 8 hours at 100° C	.8657	.4400	43.29	25.96	17.33
Bottom shelf hot-air oven, 4 hours at 125° C	.8306	.4131	41.53	24.37	17.16
Bottom shelf hot-air oven, 8 hours at 125° C	.8366	.4117	41.83	24.29	17.54
Top shelf water oven, 4 hours at 90° C	.8641	.4422	43.21	26.09	17.12
Top shelf water oven, 8 hours at 90° C	.8718	.4500	43.59	26.55	17.04
Top shelf water oven, 18 hours at 90° C	.8630	.4439	43.15	26.20	16.95
Second shelf water oven, 8 hours at 90° C	.8703	.4469	43.52	26.37	17.15
Second shelf water oven, 18 hours at 90° C	.8630	.4443	43.15	26.22	16.93
Third shelf water oven, 8 hours at 90° C	.8682	.4484	43.41	26.46	16.93
Third shelf water oven, 18 hours at 90° C	.8654	.4392	43.27	25.91	17.36
Bottom shelf water oven, 4 hours at 90° C	.8708	.4462	43.54	26.33	17.21
Bottom shelf water oven, 8 hours at 90° C	.8695	.4468	43.48	26.37	17.11
Bottom shelf water oven, 18 hours at 90° C	.8616	.4396	43.08	25.94	17.14

"I would suggest that for the sake of uniformity the time and temperature of drying be closely defined for the method; but I think my results show that the per cent of tannins is not largely influenced by variations in drying among various operators, provided they dry soluble solids and non-tannins under the same conditions at all times.

"The data I present seem to prove that the changes in weight of soluble solids and non-tannins are the same under the same conditions, and that, so far as the tannins are concerned, there is no difference in the time necessary for complete dehydration. It is only necessary, therefore, to define the time and temperature to be used."

Mr. Forbes states that after a careful investigation he found that no matter how long tanning liquor was passed through filter paper there was a slow but appreciable absorption of tanning matter; but that with an asbestos mat the absorption was nil. He communicated with the paper makers, but states that they failed to make a satisfactory paper from this material.

Mr. Forbes calls attention to the temperature of extraction of raw materials, the results shown by him in the paper read before this society last year on canaigre extraction, Bulletin No. 51, and the work of Proctor and Parker on various other materials on the same subject, and asks that this subject be given special attention.

Mr. Alsop presents the following data on the absorption of tanning matter by paper and kaolin:

A solution of gallotannin free from precipitate was continually filtered through doubled S. & S., No. 570, 15 cm filter paper, care being taken on the duplicates to have the paper weigh the same.

Original solids.	First solution, 0.8147 gram to 100 cc.		Second solution, 0.8182 gram.	
	Gram.	*Gram.*	*Gram.*	*Gram.*
First, 125 cc passing through	0.7905, loss	0.0242	0.7979, loss	0.0203
Second, 125 cc passing through	.8054, loss	.0093	8117, loss	.0065
Third, 125 cc passing through	.8077, loss	.0070	.8093, loss	.0089
Fourth, 125 cc passing through	.8085, loss	.0062	.8169, loss	.0013
Fifth, 125 cc passing through			.8166, loss	.0016
Sixth, 125 cc passing through			.8146, loss	.0036

Ten grams kaolin were then thrown on the wet paper; on the first after 500 cc had passed through, on the second after 750 cc had passed through:

Original solids.	First solution.		Second solution.	
	Gram.	*Gram.*	*Gram.*	*Gram.*
First, 125 cc	0.7996, loss	0.0151	0.8174, loss	0.0008
Second, 125 cc			.8153, loss	.0029

By filtering with 10 grams kaolin on dry filter paper:

Original solids.	First solution.		Second solution.	
	Gram.	*Gram.*	*Gram.*	*Gram.*
First, 125 cc	0.7567, loss	0.0580	0.7705, loss	0.0477
Second, 125 cc	.7969, loss	.0178	.8075, loss	.0107

By filtering a hemlock liquor, free from precipitate, under the same conditions:

Original solids.	0.6378 gram per 100 cc.	
	Gram.	*Gram.*
First, 125 cc passing through	0.6225 per 100 cc, loss	0.0153
Second, 125 cc passing through	.6299 per 100 cc, loss	.0079
Third, 125 cc passing through	.6354 per 100 cc, loss	.0024
Fourth, 125 cc passing through	.6347 per 100 cc, loss	.0031
Fifth, 125 cc passing through	.6344 per 100 cc, loss	.0034
Sixth, 10 grams kaolin added	.6179 per 100 cc, loss	.0199
Seventh, 10 grams kaolin added	.6245 per 100 cc, loss	.0133
Eighth, 10 grams kaolin added	.6388 per 100 cc, gain	.0010

Ten grams kaolin on dry paper with this liquor gave:

Original solids.	0.6378 gram per 100 cc.	
	Gram.	*Gram.*
First, 125 cc passing through	0.6023 per 100 cc, loss	0.0355
Second, 125 cc passing through	.6280 per 100 cc, loss	.0098
Third, 125 cc passing through	.6322 per 100 cc, loss	.0056

This procedure was followed on extract solutions which had been filtered clear:

	Twenty grams to liter.		Ten grams to liter.	
	Gram.	*Gram.*	*Gram.*	*Gram.*
Original solids	0.8287		0.4085	
First, 125 cc passing through	.8175, loss	0.0112	.4034, loss	0.0051
Second, 125 cc passing through	.8215, loss	.0072	.4069, loss	.0016
Third, 125 cc passing through	.8237, loss	.0050	.4081, loss	.0004
Fourth, 125 cc passing through	.8233, loss	.0054	.4100, gain	.0015
Fifth, 125 cc passing through	.8286, loss	.0001		
Sixth, 10 grams kaolin added	.8035, loss	.0252	.3895, loss	.0190
Seventh, 10 grams kaolin added	.8199, loss	.0088	.4041, loss	.0044
Eighth, 10 grams kaolin added			.4098, gain	.0013

Ten grams kaolin on paper dry.

	Twenty grams to liter.		Ten grams to liter.	
	Gram.	*Gram.*	*Gram.*	*Gram.*
First, 125 cc passing through	0.7822, loss	0.0465	0.3825, loss	0.0260
Second, 125 cc passing through	.8262, loss	.0025	.4004, loss	.0081

The variations due to two kinds of filter paper are shown by the subjoined data. No. 602 S. & S. 15 cm is a hard-finished heavy paper while No. 590 S. & S., 15 cm is a soft-finished light paper. The extract used on this was the sample of grape sugar and gallotannin sent out by the referee. The official method of filtration was pursued, and the results are given under No. 1 and under No. 2; 150 cc of solution were thrown away and then 100 cc passed through the filter and evaporated.

	Number 1.		Number 2.	
	Gram.	*Gram.*	*Gram.*	*Gram.*
Original total solids	0.7685		0.7685	
No. 590 paper	.7483, loss	0.0202	.7622, loss	0.0063
No. 602 paper	.7106, loss	.0579	.7585, loss	.0100
No. 590 paper, 10 grams kaolin	.7286, loss	.0399	.7600, loss	.0085

These results show that under the present official method a certain amount of tannin is reported as reds and that by passing 150 cc of the solution through the paper before taking the 100 cc for soluble solids most of the error due to the absorption of the paper and also of the kaolin is obviated. It also shows that different kinds of paper absorb different amounts of tanning material. It will be seen by looking over these data that the absorption by the paper is considerable, but that upon continued contact of the paper and tan liquor the paper's affinity is satisfied. The extreme slowness of filtration that ensues from passing tannin solutions through the filter until the affinity of the paper is completely satisfied makes it impractical in the method, but since the error is quite small after 150 cc have passed through, I would suggest that the method in this particular, adopted by the International Association, be embodied in the official method, except in regard to the paper. where 590 S. & S. 15 cm should be substituted for 602 S. & S. paper. This makes the directions for filtration read: "Filtrations shall take place through S. & S., No. 590, 15 cm, and the first 150 cc passing through shall be rejected. Where a clear filtrate can not otherwise be obtained, the use of about 10 grams of kaolin previously washed with a portion of the tannin solution is permissible, but is to be noted in the report." "While this does not entirely obviate the difficulty, it leaves a very small error due to this cause and seems the only practical way out."

The soluble material in the hide and the quantity of dry hide used are presented in the following table. Hide "A" is that sent out with the first sample marked "A," Vienna hide; "B" is that sent out with first sample marked "B," Ridgeway hide; "C" is that sent out with the second sample and is Vienna hide.

The samples are, No. 1, chestnut-wood extract; No. 2, hemlock liquor, and No. 3, grape sugar and gallotannin extract.

In no case does the soluble material in the hide powders exceed the limits of the method.

TABLE No. 4.—*Soluble material in and quantity of dry hide used.*

Name.	Solubles in hide.			Dry hide No. 1, sample "A" hide.	Dry hide No. 1, sample "B" hide.	Dry hide No. 2, sample "A" hide.	Dry hide No. 2, sample "B" hide.	Dry hide No. 3, sample hide.
	"A."	"B."	"C."					
	Gram.	*Gram.*	*Gram.*	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*Grams.*
Allaire	0.0070	0.0075	.....	15.43	13.98	12.99	12.55	.....
Alsop	.0025	.0030	0.0026	12.00	12.00	12.00	12.00	12.00
Carr	.0050	.0100	.....	16.50	16.50	14.75	14.75	.....
Eachus	.0020	.0035	.0020	12.00	12.50	12.00	12.50	12.00
Eitner (Weiss)	.....	.....	.....	12.42	11.97	12.42	11.97	.....
Forbes	.0030	.0028	.0025	11.48	10.14	13.05	11.89	.....
Haley	.0040	.0020	.0010	12.40	13.20	9.30	9.90	11.20
Hurty	.0039	.0041	.0066	11.37	12.23	11.77	14.39	.....
Krug	.0061	.0039	.....	.....	.....	.....	.....	.....
Kerr	.....	.....	.0065	.....	.....	.....	.....	11.15
Lowell	.0026	.0046	.....	11.73	11.95	10.56	11.83	.....
McDowell	.0070	.0090	.0030	13.92	15.12	11.84	12.60	11.55
Nichols	.....	.....	.0070	.....	.....	.....	.....	.....
Proctor	.0050	.0040	.....	10.50	12.24	12.81	13.47	.....
Small	.0026	.0022	.....	11.44	11.90	12.88	11.82	11.52
Tiffany	.0100	.0060	.....	12.35	12.50	.....	.....	.....
Yocum	.0030	.0040	.0030	12.00	11.50	11.50	11.50	12.00

In the table of results on the chestnut-wood extract the difference between maximum and minimum of solids is 1.48 per cent. This is wholly due to variations in drying. The difference in soluble solids is 3.23 per cent. Assuming that the drying variations are the same as for solids, then the variation due to filtering is 1.75 per cent. The differences on non-tannins is 2.09 per cent. On the above assumption that 1.48 per cent difference is due to drying, then 0.61 per cent is the difference in the

non-tannin determination proper. From this it is readily seen that drying and filtering are the greatest sources of variations in the tannin determination.

The hemlock liquor sent out as a sample was free from insoluble matter, yet there was reported a mean of 0.11 per cent of reds, which was undoubtedly due to an error in the method for determining them. The same thing occurs in the grape sugar—gallotannin extract, which contained no reds, yet was reported as containing on the average 1.35 per cent; the sum of the reds and tannins, 19.89, being nearly the same as the theory for tannin in this sample, 20.19 per cent. Theory gave 37.77 per cent solids, the mean of the results 38.79, a difference of 1.02 per cent, due to incomplete drying, no doubt. On non-tannins, theory gave 17.58 per cent, the mean of the results 18.90, a difference of 1.32, within 0.30 per cent the same as the difference in solids, which was due to drying entirely. From these data I think it fair to conclude that the non-tannin determination is a satisfactory one, except in regard to drying.

There seems to be no question but that the cellulose of the filter paper removed tannin to the extent of a mean of 1.35 per cent. On the other hand, in many commercial extracts a complete removal of the reds is not effected by the directions of the method for filtering, the reds being such a fine precipitate that they practically all pass through the filter. Some means of obtaining a solution free of this precipitate, and yet not having the absorption of the paper enter into the determination, must be devised before the soluble solid determination will be satisfactory. Mr. Forbes suggested an asbestos filter paper, but this can not be obtained. Mr. Alsop suggests satisfying the affinity of the paper for tannin by passing 150 cc through, and the use of kaolin, washed in the tannin solution before use, as a means of obviating both difficulties. This seems to me the only feasible means of obtaining satisfactory results, but even by this a slight error will remain.

The results of the work of Messrs. Carr and Eachus in regard to drying show the difficulties and errors of the present system. Mr. Carr's suggestion "to dry in vacuo at 70° C." would, in all probability, be the most satisfactory if it could be generally pursued, but I doubt if such a proviso could be enforced.

With this same difficulty in the determination of water in sugar solutions, sirups, etc., this association has not adopted the vacuum apparatus.

The data presented by Mr. Eachus would lead one to believe that in this determination, if the solids be exposed to decomposition in drying under the same conditions as the non-tannins, the actual loss of weight in each will be such as to give the same result in tannins.

The variations due to the various ways of drying are shown in the following table:

[Water oven, temperature, 90° C.; time, twenty-four hours. Air oven, temperature, 100° to 104° C.; time, four hours. Vacuum oven, temperature 100° C.; time, two and one-half hours; 20 inches vacuum.]

Sample.	No. 1.	No. 2.	No. 3.
	Gram.	*Gram.*	*Gram.*
Water oven	0.8241	0.7177	0.7080
Air oven	.8119	.7063	.6986
Vacuum oven	.8224	.7151	.7056

It would seem that to obtain any degree of concordance it is necessary to have the method of drying outlined with considerable exactness, both in regard to time and temperature, and it would seem to your referee a fitting subject for the forthcoming year's investigation. For the present, drying for eight hours at 100° C. in an air oven or eighteen to twenty hours in a water oven at 90° C. seems the least objectionable.

In the direct determination of water in extracts, etc., it is essential that the extract be evenly deposited as a film on the bottom of the containing vessel. This is best effected by diluting in the vessel with about 25 cc of water before evaporating. It seems better to make the directions read that 1 gram of solids will remain after drying, instead of, as now, "use 3 grams of the extract," for owing to the varying concentration of extracts sometimes the film is much too thick to dry well.

On September 30, 1897, there was organized in London the International Association of Leather Trades' Chemists, whose object is the promotion and adoption of standard methods of tannin analysis. This association received the support of practically all the Continental and English chemists interested, and after a great deal of discussion they adopted the following method for tannin:

METHOD.

PREPARATION.

Liquid extracts.—Liquid extracts shall be rapidly stirred and mixed immediately before weighing, which shall be done rapidly to avoid loss of moisture. Thick extracts which can not be otherwise mixed may be heated to 50° C., then stirred, and rapidly cooled before weighing.

Barks and solid tanning materials.—The whole sample, or not less than 250 grams, shall be ground in a mill until it will pass a sieve of five wires per centimeter. Materials such as barks, divi-divi, etc., containing fibrous material, shall be sieved and proportionate parts of the fibrous substance and that passing through sieve employed.

Strength of solution.—Such solutions shall be employed that, upon the evaporation of 100 cc the residue shall amount to between 0.6 and 0.8 gram.

Solution of liquid extracts.—A sufficient quantity shall be weighed into a covered basin or beaker, from which it shall be washed into a liter flask with about 500 cc of boiling water and well shaken, and the flask shall be filled nearly to the mark with cold water, the neck being covered with a small beaker; it shall be placed under a cold-water tap, or otherwise rapidly cooled to a temperature between 15° and 20° C. and made up to the mark, after which it shall be thoroughly mixed and immediately filtered.

Filtration.—Filtration shall take place through S. & S. filter paper No. 602. The first 150 or 200 cc of the filtrate shall be rejected. Where a clear filtrate can not be otherwise obtained, the use of a little kaolin previously washed with a portion of the tannin solution is permissible.

Extraction of solid materials.—Such quantity shall be weighed as will give an infusion of the strength already prescribed. Not less than 500 cc of the infusion shall be extracted at a temperature not exceeding 50° C., after which the temperature shall be raised to 100° C. and the extraction continued till the percolate is free from tannin and the whole made up to 1 liter, the weaker portion being concentrated if necessary by evaporation.

Total soluble matter.—One hundred cc of the clear filtered tanning solution shall be evaporated on the water bath and shall afterwards be dried in an air oven, to constant weight at a temperature of 100° to 105° C., or at a temperature not exceeding 100° C. in vacuo.

Determination of non-tannins.—The filter-tube method shall be adopted provisionally.

The Bell form of filter shall be employed. Not less than 5 grams of hide powder shall be used, and the filtrate shall be rejected so long as it gives a turbidity with a clear tannin solution. The filtrate may be used for the determination of non-tannins so long as it gives no reaction with hide-powder water (a clear solution of soluble gelatin from the hide). Fifty cc or less quantity of detannized solution shall be evaporated on a water bath and dried as directed for soluble matter.

The main difference between this and the official method of this association is in the non-tannin determination. The results from the filter tube are invariably lower than those obtained by the shaker. Messrs. Weiss, Proctor, Parker, and Touse report results on the chestnut-wood extract and hemlock liquor by this method.

	Extract.	Liquor.
	Per cent.	*Per cent.*
Weiss, nontannin	13.90	2.13
Proctor, nontannin	11.80	2.16
Parker, nontannin	12.10	2.00
Touse, nontannin	12.25	.....
Weiss, tannins	25.24	2.50
Proctor, tannins	27.90	2.45
Parker, tannins	28.00	2.80
Touse, tannins	27.91	.....

It is hoped that the more general use of the shaker method abroad will convince the foreign chemist of its superiority and that it will be incorporated in their method.

The manufacturers of extract are varying the concentration of their product much more now than in the past, and as the present method calls for 20 grams of extract per liter the solids per 100 cc of working solutions vary considerably, and inasmuch as these variations in concentration influence the results on both non-tannins and soluble solids a more definite statement regarding quantity of material is necessary.

An extract diluted 20 grams to the liter, giving 1.0648 grams of solids per 100 cc of solution, and the same extract dissolved so that 100 cc of solution had 0.8000 gram solids, gave:

	Twenty grams to liter.	0.8 gram per 100 cc.
	Per cent.	*Per cent.*
Soluble solids	50.00	51.15
Non-tannins	19.44	18.46

Uniformity in the quantity of hide and strength of solution are both necessary for concordance in the results on non-tannins, as has been proven, and with the absolute definition of the quantity of solids per 100 cc of solution and the use of a certain quantity of hide powder this error is eliminated.

The directions for washing the hide powder as a preliminary to the non-tannin determination are quite complicated and lengthy. To determine if some better means could not be devised several experiments were carried on, and it was found that by using about twenty-five times as much water as hide, by weight, a very satisfactory washing could be effected by one treatment.

Twenty grams of hide and 500 cc of water were used in the following tests: the hide was well stirred in a large beaker, fresh water was used, and the hide and water stood one hour for each washing:

	Gram.
First washing, 100 cc, evaporated	0.1014
Second washing, 100 cc, evaporated	.0070
Third washing, 100 cc, evaporated	.0061
Fourth washing, 100 cc, evaporated	.0079

If the hide answer the tests outlined under Paragraph VIII of the method, the preparatory washing can be much simplified and one of the most tiresome and objectionable points of the method obviated.

RECOMMENDATIONS.

Your referee makes the following recommendations:

That Paragraph II of Method, "Quantity of material," shall read:

"In the case of bark and similar material use such quantity as will give about 0.8 gram solids per 100 cc of solution, extract in Soxhlet or similar apparatus at steam heat for non-starchy materials. For canaigre and substances containing like amounts of starch use temperature of 50° to 55° C. until near complete extraction, finishing the operation at steam heat. In the case of extract weigh such quantity as will give 0.8 gram solids per 100 cc of solution, dissolve in 900 cc of water at 80° C., let stand twelve hours, and make up to 1,000 cc."

That Paragraph III shall read:

"Weigh 2 grams if it be an extract into a flat-bottomed dish not less than 2½ inches in diameter; add 25 cc of water, warm slowly till dissolved; continue evaporation and dry."

That the following be inserted as IIIa:

"All dryings called for, after evaporation to dryness on water bath or others, shall be done by one of the following methods, the soluble solids and non-tannins being dried under similar, and so far as possible, identical conditions:

"A. For twenty-four hours at the temperature of boiling water in a steam bath.

"B. For eight hours at 100° to 103° C. in an air bath.

"C. To constant weight in vacuo at 70° C.

"State in report the method of drying pursued."

That Paragraph V shall read:

"Filtration shall take place through a double folded filter (S. & S. No. 590), the first 150 cc passing through shall be rejected, 100 cc next passing through shall be evaporated and dried. When a clear filtrate can not be otherwise obtained the use of 10 grams of kaolin previously washed in a portion of the tanning solution is permissible. Evaporation during filtration must be guarded against."

That Paragraph VI shall read:

"Prepare 20 grams of hide by washing in a No. 7 beaker (Griffin) with 800 to 1,000 cc of water, stir well and let stand one hour; filter the magma through linen, squeeze thoroughly by hand and remove as much water as possible by means of a press, etc." (From here the same as in Bulletin No. 51.)

That the following be inserted as VIIa:

"A. That the temperature of solutions shall be between 16° and 20° C. when measured or filtered.

"B. Dryings to be made in flat-bottomed dishes not less than 2½ inches in diameter."

No papers were presented on the subject of tannin.

The committee on recommendations here presented its report in favor of the adoption of the recommendations of the referees on food adulteration and tannin. The report was adopted.

Dr. De Schweinitz then read his report on the determination of nicotin, as follows:

REPORT OF AN INVESTIGATION OF THE METHODS FOR THE DETERMINATION OF NICOTIN IN TOBACCO POWDERS AND EXTRACTS.

By E. A. DE SCHWEINITZ, with the cooperation of J. A. EMERY and F. K. CAMERON.

Having had occasion to examine the nicotin content of a number of tobacco extracts, my attention was called to the fact that by the best method, namely the Kissling, which is ordinarily used, the results are not always concordant.

It occurred to me, therefore, that it might be of considerable interest to have a number of different men make analyses of the same samples. I wrote to Dr. Winton,

president of the association, in regard to the matter, and he said that I should proceed with the work and agreed to cooperate in it. Accordingly, I secured samples of two tobacco powders and two tobacco extracts from Dr. Walker Bowman, of the Louisville Spirit Cure Tobacco Company, and had duplicate samples bottled and sent to the following gentlemen who had agreed to aid in the work: Prof. R. J. Davidson, of Blacksburg, Va.; Prof. J. H. Patterson, Maryland Agricultural College; Dr. A. L. Winton, of Connecticut, and Dr. Walker Bowman, of the Louisville Spirit Cure Tobacco Company, and in my laboratory the work was done by Mr. James A. Emery and Dr. Frank K. Cameron. Three methods were prescribed, namely, the Kissling, the use of Lloyd's reagent, and the optical method. All of the gentlemen carried out the work according to Kissling's method, but only two or three used the optical method or Lloyd's reagents.

The outlines of the several methods used are as follows:

Kissling's Method.[1]—Extraction of the alkaline magma with ether evaporation, distillation, etc.

The use of Lloyd's reagent.—The outline of this method is as follows: Five cc of the solution or extract are placed in an extra heavy beaker and enough of Lloyd's reagent for alkaloids (equal parts dry ferric hydrate and sodium bicarbonate) is then added to make a very stiff magma or paste. Twelve cc of rectified petroleum ether are then added to the contents of the beaker, and the whole rapidly agitated. The petroleum ether is then decanted into a small separating funnel and the operation repeated upon the pasty mass with 11, 10, 9, and 8 cc of ether in turn. The ether extracts are mixed together in the separating funnel and 5 cc of normal sulphuric acid with 15 cc of water added to the contents. The whole is rapidly shaken and the lower layer withdrawn. The ether is again washed with 10 cc and 5 cc of water and the washings, together with the acid extract, titrated with one-fifth normal potassium hydrate, litmus being added as an indicator. Each cubic centimeter of alkali used in excess of that necessary to neutralize the acid is equal to 0.0324 gram nicotin.

The optical method.—The optical method described by Popovici (Ztschr f. Physiol. Chemie, Vol. XIII, p. 445; also Chem. Ztg. 1889, p. 1030). The outline of the method is as follows:[2]

The tobacco is extracted with ether as in Kissling's method, and the extract from 20 grams of tobacco is mixed with 10 cc of a fairly concentrated nitric acid solution of phosphomolybdic acid, which throws down the nicotin, ammonia, and so on, as a precipitate which separates readily. The ether is then carefully poured off and the residue is made up to a volume of 50 cc by the addition of distilled water. Eight grams of finely powdered barium hydrate are then added for the purpose of setting the nicotin free. As this decomposition takes place somewhat slowly, the flask containing the mixture should be allowed to stand for several hours and be shaken from time to time. The precipitate, which is at first blue, gradually changes to a blue-green, and finally to a yellow. The alkaline solution which contains the nicotin is filtered off and the yellow-colored filtrate is polarized. The reading on the polariscope in minutes is noted. From this reading, in combination with the table which can be found in Kissling's Tabakkunde, page 68, the percentage of nicotin can be determined. The results reported by the different gentlemen who carried out the work can be seen in the following table:

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[1] Wiley, Principles and Practice of Agricultural Analysis, Vol. III, p. 606.

[2] Vide Principles and Practice of Agricultural Analysis, Vol. III, p. 607.

Tobacco powders.

Analyst and location.	No. 1.				No. 2.			
	Kissling.	Lloyd.	Optical.	Average.	Kissling.	Lloyd.	Optical.	Average.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
A. L. Winton, Connecticut	0.70	.....	.....	.....	1.67	.....	.....	K=1.70
				K=0.67	1.73			
	.65							
H. J. Patterson, Md. Agr. Ex. Sta.	.53	.....	.....	K= .53	1.36	.....	.....	K=1.36
R. J. Davidson, Va. Agr. Ex. Sta.	.81	0.90	1.42	K= .81	1.85	1.94	.....	K=1.83
	.81	.....	.....	L= .90	1.81	1.81	.....	L=1.87
				O=1.42				
Walker Bowman, Louisville, Ky.	.77	.....	.....	K= .77	2.52	.....	.....	K=2.52
J. A. Emery, U. S. Biological Chemical Laboratory.	.58	.50	.....	K= .58	2.56	2.05	1.75	K=2.56
				L= .50	.....	.....	.....	L=2.05
								O=1.75

Tobacco extracts.

Analyst and location.	No. 3.				No. 4.			
	Kissling.	Lloyd.	Optical.	Average.	Kissling.	Lloyd.	Optical.	Average.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
A. L. Winton, Connecticut	3.60	.....	.....	K=3.64	12.66	.....	.....	K=12.76
	3.64	.....	.....	.....	12.87			
	3.68							
H. J. Patterson, Md. Agr. Ex. Sta.	3.05	.....	.....	K=3.05	9.92	.....	.....	K=9.92
R. J. Davidson, Va. Agr. Ex. Sta.	3.93	4.14	4.13	K=3.95	11.73	11.21	10.90	K=11.75
	3.98	3.95	4.00	L=4.02	11.77	9.91	.....	L=10.56
				O=4.06	.....	.....	.....	O=10.90
Walker Bowman, Louisville, Ky.	4.24	.....	.....	K=4.24	9.11	.....	.....	K=9.11
F. K. Cameron, U. S. Biological Chemical Laboratory.	2.50	.....	.....	.....	10.80			
	2.50	.....	.....	.....	11.50			
	2.52	.....	.....	K=2.54	11.06			
	2.76	.....	.....	.....	14.40	.....	.....	K=12.36
	2.90	.....	.....	.....	11.40			
	2.20	.....	.....	.....	15.10			
	2.40	.....	.....	.....	11.60			
					13.07			
J. A. Emery, U. S. Biological Chemical Laboratory.	3.91	3.88	.....	K=3.91	14.70	13.35	10.00	K=14.70
		3.80	.....	L=3.84	.....	13.38	.....	L=13.40
						13.47	.....	O=10.00

The very great variation in these results from the same samples in the hands of different men made me think that a still more extensive study than that which I had intended originally was desirable. Accordingly, Mr. James A. Emery and Dr. Frank K. Cameron undertook a very thorough investigation of the subject. We have tried all manner of modifications of the ordinary methods and a large number of new methods, that will be noted below, without having been able as yet to hit upon a method which would be surely accurate. The sources of error in the Kissling method were found to be due to ammonium salts and the amins, which it is very difficult to separate from nicotin on account of the fact that the salts which all three of these form are so nearly alike. We note briefly some of the different methods which we have tried to adapt to a more careful separation of the nicotin from ammonium salts and amins.

THE FORMATION OF AN INSOLUBLE PRECIPITATE.

So far none has been found to which there are not serious objections as a practicable analytic method. The picrate is quite measurably soluble. The phosphotungstate, phosphomolybdate, etc., are too nearly alike in their general physical properties to the corresponding salts of other organic nitrogenous bases to make a satisfactory separation possible. The insoluble double salts with metallic bases are dependent on conditions of temperature and concentration to such an extent as to make the precipitation of any definite compound too uncertain under ordinary working conditions. Some of these objections will be further discussed presently.

ROTATORY EFFECT OF SOLUTIONS ON PLANE OF POLARIZED LIGHT.

An elegant method depending on this principle has been suggested by Popovici; but the possibility of errors incident to the preparation of the solutions is great. The presence of optically active solutes other than nicotin is not considered. For these or other possible reasons this method has not found favor with analysts. A further minor objection, but one greatly to be regretted, is that few laboratories have the necessary instrumental equipment.

PRECIPITATION WITH PICRIC ACID.

Preliminary experiments with tobacco extracts having indicated the possibility of this method being used, the following experiments were made:

Experiment 1.

To solution known to contain nicotin, 0.52245 gram.

Added 10 cc N/2 HCl.

Added saturated solution of picric acid to excess.

Heated to 98° C., then cooled slowly to room temperature and allowed to stand twenty-four hours.

Filtered precipitate and washed with 100 cc water.

Weight of precipitate, 1.79700 grams.

By calculation [$C_{10}H_{14}N_2$—$2C_6H_2(NO_2)_3OH$] gave nicotin 0.4695 gram, or 89.86 per cent recovered.

The precipitate was dissolved in 20 cc N/2 NaOH, distilled in current of steam into 41 cc N/2 HCl; about 1,000 cc in distillate.

	Cubic centimeters.
41 cc N/2 acid were equivalent in alkali to	205.00
Distillate equivalent in alkali to	170.40
Nicotin equivalent in alkali to	34.60

34.6 cc alkali were equivalent in acid N/2 to 6.92 cc.

34.6 cc alkali were equivalent in nicotin to 0.56052 gram, or 107.28 per cent recovered.

The filtrates from nicotin picrate and washings were made alkaline with soda and distilled with steam into 20 cc N/2 acid, about 700 cc in distillate.

	Cubic centimeters.
20 cc N/2 acid were equivalent in alkali to	100.00
Distillate	98.50
Nicotin	1.5
1.5 cc alkali were equivalent in N/2 acid to	.30
	Gram.
1.5 cc alkali were equivalent in nicotin to	0.0243
Nicotin from precipitate (original)	.4695
Nicotin from filtrate	.0243
	.4938

Percentage recovered, 94.51.

Nicotin from distillate (original)	.56052
Nicotin from filtrate	.02430
	.58482

Percentage recovered, 111.97.

Experiment 2.

	Gram.
Solution known to contain nicotin	0.74196
Ammonia	.08500
Trimethylamin	(?)

Added H_2SO_4 solution to slight excess. Then a saturated solution of picric acid to excess. Heated until precipitate was entirely dissolved and then cooled gradually, finally standing the containing flask in an ice-water bath.

Filtered and washed with 150 cc water, in three portions, sucking dry each time with the aid of the pump. Dried precipitate at 120°.

Weight of precipitate, 2.6708 grams.

By calculation [$C_{10}H_{14}N_2$—2 $C_6H_2(NO_2)_3OH$] gave nicotin 0.6934 gram, or 93.4 per cent recovered.

The filtrate was brought into a flask with a rather large excess of soda, and distilled in a current of steam into 30 cc N/2 HCl and titrated back with soda.

Distillate was equivalent in N/2 acid to 12.52 cc.

Nicotin in distillate was equivalent to 1.01412 grams, or 136.7 per cent recovered.

From these experiments (1 and 2) it would appear that either all the nicotin was not precipitated, or that nicotin picrate is decidedly more soluble than the rather vague statements in the literature would indicate. Therefore the following experiments were made:

Experiment 3.

[Solubility of nicotin picrate at 20.5°.]

	Grams.
Solution containing nicotin picrate weighing	9.9723
Evaporated to dryness, nicotin picrate weighing	.0055
Water	9.9668

One gram of water contained 0.0005519 gram nicotin picrate.

Solubility (uncorrected) of nicotin picrate in water, 0.5519 gram per liter.

Experiment 4.

	Grams.
Solution containing nicotin picrate weighing	9.9774
Evaporated to dryness, nicotin picrate weighing	.0055
Water	9.9719

One gram of water contained 0.0005515 gram nicotin picrate.

Solubility (uncorrected) of nicotin picrate in water is 0.5515 gram per liter.

Average solubility: 1 part of nicotin picrate in 1,813 parts of water.

In experiments 1 and 2 about 500 cc of water for solution and washings were used. A loss of 0.25 gram, approximately, could therefore be expected.

For experiment 1, actual loss in percentage, 10 per cent; calculated, 14 per cent.

For experiment 2, actual loss in percentage, 6.5 per cent; calculated, 9 per cent in round numbers. It was thought desirable to determine with approximate accuracy the probable loss from washings of the precipitate, therefore the following experiments were made:

Experiment 5.

Precipitated 16 cc nicotin solution and weighed in a gooch.

	Grams.
Weight of nicotin picrate and gooch	27.5075
Weight of gooch	25.4194
Weight of nicotin picrate	2.0881

16 cc nicotin solution equal 7.36 cc N/2 HCl equal 0.59616 gram nicotin; 2.2815 grams nicotin picrate.

2.0881 grams nicotin picrate equal 0.54562 gram nicotin; 91.52 per cent nicotin recovered.

0.1934 gram nicotin picrate was lost in solution.

8.47 per cent nicotin picrate were lost in solution.

Experiment 6.

Took No. 1 gooch and added crystals of nicotin picrate crystallized from hot solution.

	Grams.
Weight of nicotin picrate and gooch	26.9080
Weight of gooch	26.7107
Weight of nicotin picrate	0.1973

Washed with aid of vacuum pump with 250 cc distilled water at temperature of 23° C., filling the gooch each time and then sucking dry before adding more water, dried and weighed.

	Grams.
Weight of nicotin picrate and the gooch	26.8761
Weight of gooch	26.7107
Weight of nicotin picrate	0.1654

16.22 per cent were lost by washing.

It follows from the above results that the estimation of nicotin as picrate is open to grave objections, as under the usual conditions of work a control of the amount lost in the washing is quite impracticable. It follows, further, from experiments 1 and 2, that a quantitative recovery of the nicotin from the picrate, which it was hoped would serve as a check, is not possible, at least by distilling with steam from an alkaline solution. The contents of the flask from which the distillation was making gradually became darker and darker, and the experiments quoted, together with others which we made, showed that very great decomposition took place, depending on the time of heating and the concentration of the flask contents.

PRECIPITATION WITH PHOSPHOMOLYBDIC AND PHOSPHOTUNGSTIC ACIDS.

Pyridin and the aliphatic amins were found to yield precipitates so nearly identical in physical properties, conduct toward solvent, etc., with those from nicotin that all attempts to found a method of separation upon them were unavailing.

PRECIPITATION AS DOUBLE SALTS WITH METALLIC BASES.

In all cases to which reference could be found in the literature, it was evident that the salt in question was one of a series, and that in practice the composition of the precipitate would be too uncertain to be of value. No experiments were made in this direction.

PRECIPITATION WITH BROMIN AND IODIN.

In neutral or acid solution nicotin forms insoluble addition products with bromin and iodin. Repeated efforts were made to utilize these facts, but the precipitates were in all cases gummy or pasty, so filtration was impracticable. Working in ether as a solvent no better results could be obtained. The precipitation of the nicotin with bromin and washing it free from other substances by decantation was tried. The nature of the precipitate made the process itself a rather difficult one; but farther, the precipitate decomposed on standing or heating alone, with water, or with alkaline solutions, with the formation of a greater or less amount of tar. The recovery of the nicotin quantitatively was impracticable.

Of the methods in use the one which has found most favor with analysts, and justly so, is known by the name of its author as the Kissling. Briefly, it consists in mixing intimately the material with alcoholic potash, the extraction of the nicotin with some suitable solvent (ether is generally recommended), removal of the solvent by evaporation at as low a temperature as practicable, and separation of the nicotin from the residue by distilling from an alkaline solution in a current of steam. A systematic examination of this method was deemed advisable, therefore the following experiments were made.

The extraction of the material was conducted in the following manner: An apparatus was arranged, consisting of a glass tube with a cross section about 45 mm in diameter, about 225 mm in length, drawn out to a narrow tube at the bottom, and fitted into a small Florence flask, about 250 cc capacity, with a well-fitting rubber stopper. The larger end was closed with a rubber stopper carrying a long condenser. The material to be extracted was placed in a cone or cylinder, made by folding a sheet of filter paper over a cylinder of wood or some suitable material, rolling it until of desirable thickness, and then holding it in position by tying with a piece of thin wire. This cylinder was then placed in the tube just mentioned. The paper cylinder may be kept from stopping the tube by first introducing a piece of broken glass or similar material, but our experience showed this precaution to be required but very seldom. When properly arranged, this is a most rapid and effective instrument for continuous extraction at a temperature but little below the boiling point of the solvent, and has the advantage of simplicity and that it can be constructed by anyone with usual laboratory material. When liquid extracts were under investigation they were first absorbed by precipitated calcium or magnesium carbonate and then treated in the usual manner.

Experiment 9.

Some qualitative experiments were made to test the value of ether, benzene, ligroin, and alcohol as solvents for extraction. Their desirability for the purpose was found to be in the order named, with a very decided preponderance in favor of the first mentioned. A further description of these experiments seems hardly worth while.

Experiment 10.

Some odor of nicotin was found to be present in material which had been subjected to extraction as above described, therefore extractions were made of widely varying amounts of material for a certain number of hours. The operation was discontinued, a fresh quantity of solvent introduced, and the contents of the second extract estimated. The results are here tabulated:

Solvent.	First extraction.	Second extraction.	Second extraction.
	Hours.	*Hours.*	*Per cent.*
Ligroin	12	6	0.016
	12	6	0004
Benzene	3	6	0000+
	3	6	0000+
Ether	3	6	0000+
	3	6	0000+

It follows that no appreciable error is to be expected from this source.

We next tested the extraction of nicotin by ether, to find out the probable error introduced by the evaporation of the solvent. We had been using the device of acidifying the extract before driving off the ether, but the result of the following experiment showed this to be an unnecessary refinement and further had the disadvantage of retaining ammonia and other very volatile basic constituents which would have been more or less completely removed with the ether.

Experiment 11.

Started with nicotin (Merck) 1.4186 grams dissolved in 25 cc ether (c. p. Squibb).
Evaporated slowly. Dissolved residue in water and made up to 100 cc.
Added 40 cc N/2 HCl and titrated, requiring 39.4 cc soda solution.

	cc
40 cc N/2 acid are equivalent in soda	68.92
Solution equivalent in soda to	39.40
Nicotin equivalent to	29.52

29.52 cc alkali equivalent to N/2 HCl 17.13 cc.
29.52 cc alkali equivalent to nicotin 1.3875 grams, or 97.74 per cent, recovered
Checked with the following experiment:
Started with nicotin (Merck.) 0.9042 gram made up to 100 cc in water.

Added 20 cc N/2 acid equivalent to	34.46 cc alkali.
Nicotin + N/2 acid equivalent to	15.60 cc alkali.
	18.86 cc alkali.

18.86 cc alkali are equivalent to 10.94 cc N/2H_2SO_4.
18.86 cc alkali are equivalent to 0.8860 gram nicotin.
97.99 per cent nicotin in original substance.
Loss by extraction with ether and evaporation, 0.25 per cent.

The efficiency of distillation in a current of steam as a method of recovering the nicotin was then tested by the following experiment:

Experiment 12.

Started with solution containing nicotin, 1.3867 grams.
Added 15 cc alkali [1.725 alk = 1 cc N/2 acid].
Distilled 400 cc into 30 cc N/2 H_2SO_4.

30 cc N/2 H_2SO_4	51.69 cc alkali.
30 cc N/2 H_2SO_4 and nicotin in distillate	26.25 cc alkali.
Nicotin equivalent to	25.44 cc alkali.

25.44 cc alkali equivalent to 14.75 cc N/2 acid.
25.44 cc alkali equivalent to 1.1948 grams nicotin.
86.1 per cent of nicotin recovered.
Continued distillation, 400 cc into 10 cc N/2 H_2SO_4.

10 cc N/2 H_2SO_4 required alkali	17.23	cc
10 cc N/2 H_2SO_4 plus nicotin	13.20	
Nicotin equivalent to	4.03	cc
4.03 cc alkali equivalent to N/2 H_2SO_4	2.34	
4.03 cc alkali equivalent to nicotin	0.1893	gram

99.81 per cent nicotin recovered.

The practice here differed from that recommended by Kissling. According to his directions the distillation should be so conducted that the contents of the flask from which the distillation is taking place should rapidly diminish in volume. But experience led us to believe this bad practice, on account of the probable decomposition of the nicotin and other organic nitrogen bases present in the more concentrated alkali. The above-described experiment shows that a much larger volume must be distilled if the contents of the distillation flask are to be kept approximately constant. We believe the latter practice to be much the more desirable.

This experiment was repeated, starting with nicotin and ammonia.

Experiment 13.

Took solution containing nicotin 1.8826 grams and 100 cc solution of ammonia, equivalent to 35 cc N/2 H_2SO_4.

Added 50 cc soda solution [1.723 cc alk. = 1 cc N/2 acid].

Distilled 800 cc into 100 cc N/2 H_2SO_4 and titrated with standard alkali.

100 cc N/2 H_2SO_4 are equivalent to	172.30 cc soda.
Distillate is equivalent to	85.50 cc soda.
Nicotin and ammonia are equivalent to	86.80

86.80 cc alkali are equivalent to 50.38 cc N/2 H_2SO_4.

Original solution before distillation required 51 cc N/2 H_2SO_4.

98.79 per cent volatile alkali recovered. That is to say, that practically all the ammonia, as well as nicotin, was recovered. But the following experiments indicate that the ammonia is effectively eliminated in the evaporation of the solvent from the ether extract. The method employed is that of Kissling with these modifications—that the contents of the distilling flask are kept at an approximately constant volume and the distillate is always acid.

Experiment 14.

To 10 cc nicotin solution, containing 0.35721 gram nicotin, added 10 cc N/2 NH_3 and 5 cc of alcoholic soda (made up per Kissling) with $CaCO_3$; extracted with ether in continual extraction apparatus; evaporated ether and made alkaline with soda solution and distilled with steam into flask containing 20 cc N/2 HCl until 400 cc distillate had collected; then put into flask containing 10 cc N/2 HCl and continued until 400 cc more had distilled over.

20 cc N/2 HCl equivalent to	100	cc N/10 soda.
20 cc N/2 HCl plus nicotin equivalent to	78.8	cc N/10 soda.
Nicotin equivalent to	21.2	cc N/10 soda.
10 cc N/2 HCl equivalent to	50.0	cc N/10 soda.
10 cc N/2 + nicotin equivalent to	49.3	cc N/10 soda.
	.7	cc N/10
	21.2	cc N/10
	21.9	cc N/10

= 4.38 cc N/2 HCl, or 99.32 per cent nicotin recovered.

Experiment 15.

Started with solution containing nicotin 0.1786 gram (5 cc N/2 NH_4OH solution).
5 cc alcoholic soda (Kissling) absorbed with sufficient calcium carbonate.
Extracted with ether 6 hours.
Evaporated ether, dissolved residue in 50 cc N/10 NaOH and distilled 700 cc in 25 cc N/2 HCl.

25 cc N/2 acid equivalent to	125 cc N/10 alkali.
Distillate equivalent to	114 cc N/10 alkali.
Nicotin equivalent to	11 cc N/10 alkali.

11 cc N/10 alkali equivalent to 2.2 N/2 acid.
11 cc N/10 alkali equivalent to 0.1782 gram nicotin.
99.78 per cent were recovered.

Experiment 16.

Solution containing nicotin 0.35721 gram and 5 cc N/2 NH_4OH solution and 5 cc alcoholic solution NaOH (Kissling). Absorbed mixture with sufficient calcium carbonate. Extracted with ether for three hours. Evaporated ether. Made alkaline. Distilled 700 cc into 30 cc N 2 HCl, and titrated back with alkali solution.

	cc.
30 cc N/2 acid equivalent to	30.3
Distillate equivalent to	26.0
Nicotin equivalent to	4.3

97.5 per cent nicotin recovered.
These results were satisfactory. But when trimethylamin was present in the original solution it was not removed by the process, and, judging from the results of the next experiment, the removal of ammonia was also very much less effective.

Experiment 17.

Solution containing nicotin 0.18549 gram and 5 cc trimethylamin hydrochlorid (dilute solution, strength unknown) and 5 cc N/2 NH_4OH solution and 5 cc alcoholic soda (Kissling).
Absorbed with powdered $MgCO_3$; extracted with ether for upward of three hours. Evaporated extract. Dissolved residue in 50 cc N/10 NaOH solution.
Distilled 800 cc with steam into 30 cc N/2 HCl.

30 cc N/2 HCl equivalent to	150.00 cc N/10 NaOH
Distillate equivalent to	136.80
Nicotin equivalent to	13.20

13.2 N/10 NaOH equivalent to 2.64 N/2 HCl.
13.2 N/10 NaOH equivalent to 0.21384 gram nicotin.
115.28 per cent nicotin were recovered.
An attempt was made to correct this determination by the following experiment:

Experiment 18.

Solution containing 10 cc trimethylamin hydrochlorid (dilute solution, strength unknown); 10 cc N/2 NH_4OH solution; 5 cc alcoholic soda (Kissling).
Absorbed with powdered magnesium carbonate.
Extracted with ether for upward of three hours. Ether was evaporated slowly, residue taken up with 50 cc N/10 NaOH.
Distilled 400 cc (with current of steam) into 20 cc N/2 HCl.
Titrated back with N/10 NaOH.

20 cc N/2 HCl are equivalent to	100 cc N/10 NaOH
Distillate equivalent to	98 cc
Volatile alkali is equivalent to	2 cc

2 cc N/10 are equivalent to 0.4 cc N/2 HCl.
2 cc N/10 are equivalent to 0.0324 gram nicotin.

Correction for experiment 17	0.01620 gram nicotin
	.21384 gram nicotin
Corrected	0.19764 gram nicotin, or

106.55 per cent nicotin were apparently recovered. This indicates a serious defect in the method at this point.

Attempts were now made to eliminate the bases other than nicotin by decomposing them. Nitrous acid does not, strange to say, form a nitroso compound with nicotin, in spite of the generally accepted constitutional formula assigned to that compound. Primary amins should be removed by decomposition and elimination of their nitrogen. Secondary amins should yield easily separated nitroso compounds, and by boiling in neutral solutions the ammonia should decompose entirely into nitrogen and water. A large number of careful experiments was tried with these objects in view. The results were disappointing. Tobacco extracts were found to contain relatively large proportions of trimethylamin, and this being a tertiary amin was unaffected by nitrous acid. Ammonium nitrite was found to decompose on boiling at a surprisingly slow rate. After four hours boiling the solution was found to contain a large percentage of undecomposed ammonia. This is now the subject of an independent investigation. Finally the following experiments will show that the process was not without effect on the nicotin itself. A blank was run first.

Experiment 19.

To 10 grams $NaNO_2$, added 30 cc H_2SO_4 (3/N?).
Slowly heated to boiling. Boiled until all acid fumes were removed. Made alkaline with NaOH solution and distilled into water colored with cochineal. Very slight evidence of alkaline reaction.

Experiment 20.

Started with solution containing 0.365 gram nicotin.
Added 20 cc N/2 H_2SO_4.
Added 5 grams $NaNO_2$.
Allowed to stand one hour in cold with occasional shaking.
Added 50 cc alkali [1.723 cc alk. = 1 cc N/2 acid.]
Distilled with steam until about 300 cc had come over, distilling into 30 cc N/2 H_2SO_4.

30 cc N/2 H_2SO_4 equivalent to	51.69 cc alkali
Distillate equivalent to	44.50 cc alkali
Nicotin equivalent to	7.19 cc alkali

7.19 cc alkali are equivalent to 4.17 cc N/2 H_2SO_4.
7.19 cc alkali are equivalent to 0.3377 gram nicotin.
92.5 per cent nicotin were recovered.

Experiment 21.

Started with solution containing nicotin 0.3547 gram.
Added 20 cc N/2 H_2SO_4.
About 5 grams $NaNO_2$.
Stood one hour.
Made alkaline with 50 cc NaOH solution [1.723 cc alk. = 1 cc N/2 acid.]
Distilled with steam (until about 300 cc came over) into 30 cc N/2 H_2SO_4.

30 cc acid equivalent to	51.69 cc alkali
30 cc acid and nicotin equivalent to	45.90 cc alkali
Nicotin equivalent to	5.79 cc alkali

5.79 cc alkali are equivalent to 3.36 cc N/2 H_2SO_4.
5.79 cc alkali are equivalent to 0.2722 gram nicotin.
76.7 per cent nicotin recovered.

Experiment 22.

Started with solution containing nicotin 0.7171 gram.
Added 18 cc acid N/2 H_2SO_4.
Added 10 grams $NaNO_2$ and allowed to stand over night.
Made alkaline with 50 cc soda solution, [1.723 alk. = 1 cc N/2 acid.]
Distilled 800 cc into flask containing 60 cc N/2 H_2SO_4.

60 cc N/2 H_2SO_4 equivalent to	103.38 cc alkali
60 cc N/2 H_2SO_4 and nicotin equivalent to	89.50 cc alkali
Nicotin equivalent to	13.88 cc alkali

13.88 cc alkali equivalent to 8.03 cc N/2 H_2SO_4.
13.88 cc alkali equivalent to 0.6521 gram nicotin.
90.93 per cent nicotin were recovered.
Continued distillation, 400 cc into flask containing 10 cc N/2 H_2SO_4.

10 cc N/2 H_2SO_4 equivalent to	17.23 cc alkali
10 cc N/2 H_2SO_4 and nicotin equivalent to	17.23 cc alkali

No nicotin in distillate.

Apparently the nicotin is oxidized more or less, depending on the conditions. The attempt was made next to get rid of the ammonia, etc., by treatment with sodium hypobromite and calcium hypochlorite, but decomposition of the nicotin made further attempts to apply the method useless. Further, the necessity of working in alkaline solutions made a large error by loss of nicotin very probable. It was not possible, of course, to acidify the solutions for the purpose of transferring them, etc., in view of the experiment with bromin, referred to above.

Ammonium oxalate is insoluble in alcohol, and comparatively so in aqueous alcohol, if the relative amount of water present be not large. Nicotin oxalate and the oxalates of organic bases generally are quite soluble. In order to test this as a method of separation, the solubility of the ammonium oxalate was determined in the following experiments:

Experiment 23.

Solution of ammonium oxalate in 99 per cent alcohol.

Saturated solution of ammonium oxalate in alcohol at 17.5° C	79.6365 grams
Evaporated, residue weighed	9.0097 grams
Solvent weighed	79.6348 grams

Equivalent to 0.0169 gram ammonium oxalate in a liter, or 1 part ammonium oxalate is soluble in 46,844 parts 99 per cent alcohol at 17.5° C.

Experiment 24.

Solution of ammonium oxalate in 78 per cent alcohol.

Saturated solution at 18.50 weighed	79.2150 grams
Evaporated, residue weighed	0.0177 gram
Solvent weighed	79.1973 grams

Equivalent to 0.18 gram in 1 liter, or 1 part ammonium oxalate is soluble in 4,475 parts 78 per cent alcohol. In view of these results the attempt was next made to separate ammonia from nicotin, with the following results:

Took solution of nicotin in water equivalent to 12 cc N/2 HCl.
Took solution of ammonia equivalent to 8 cc. Volume of mixture about 35 cc.
Added 98 per cent alcohol, about 350 cc, and a saturated solution of oxalic acid in alcohol until marked acid reaction. The whole was then cooled with ice water,

being shaken from time to time. The ammonium oxalate formed was then separated by filtration, with aid of the pump, and washed with 98 per cent alcohol. The filtrate and washings were then evaporated to small volume, the residue made up to about 400 cc with water, the solution made slightly alkaline and distilled in a current of steam. The distillate required 11.82 cc N/2 HCl to neutralize, or 98.5 per cent nicotin were recovered. This experiment was repeated. Distillate required, 11.9 cc N/2 HCl to neutralize it, or 99.1 per cent nicotin were recovered.

Estimation of nicotin in a tobacco extract which also contained much ammonia:

	Per cent.
By treatment with sodinm nitrite	14.60
By treatment with sodium nitrite	18.44
By treatment with sodium nitrite	9.66
By treatment with sodium nitrite	10.64
By treatment with sodium nitrite	8.57
By treatment with sodium nitrite	17.88
By Kissling	13.59
By Kissling	12.46
Separation of ammonia as oxalate	5.95
Separation of ammonia as oxalate	6.00

The following conclusions seem warranted by the facts just presented:

I. Up to the present time no reliable method for the separation of nicotin from ammonia and other organic bases by precipitation has been suggested.

II. A complete extraction of nicotin by ether and some other solvents is readily accomplished.

III. An evaporation of an ether extract affords a practicable separation from ammonia alone, but not from other organic bases.

IV. In a separation by distillation with a current of steam, it is preferable to keep the volume in the distillation flask approximately constant. Much larger distillate is required in order to effect a complete separation than is usually supposed.

V. No method of separation by decomposition of accompanying bodies has been found practicable.

VI. A separation of nicotin from ammonia by means of alcoholic oxalic acid can be effectively accomplished.

VII. The presence of tertiary amins, and possibly some pyridin derivatives in tobacco extracts, is as yet an insurmountable difficulty in the separation or estimation of nicotin.

Professor Winton also reported the following modification of the Kissling method:

A METHOD WITHOUT EXTRACTION.

I devised this method with the idea that for commercial purposes Kissling's process, in the case of extracts, could be considerably shortened. The details are as follows: From 1 to 5 grams of the extract are weighed into a 400 cc flask having a mouth 3 cm in diameter. If the solution be so thick as not to flow easily it is diluted with 1 cc of water. One cc of a merely saturated solution of caustic soda is then added and by gentle agitation mixed as completely as possible with the extract. The following experiments indicate that all the ammonia is removed after standing without corking at the room temperature for twenty-four hours. Two 1 cc portions of 30 per cent ammonia treated by this process with 1 cc of soda yielded after standing twenty-four hours, on distillation with 50 cc N/10 caustic soda, distillates which neutralized respectively 0.15 and 0.20 cc N/10 sulphuric acid. A solution of pure nicotin gave by this method 12.66 per cent and 13.17 per cent nicotin, while a weighed portion of the same solution mixed with 1 cc 30 per cent ammonia gave 12.91 per cent. In the table are given results after standing with the strong soda, 24, 48, 72

and 100 hours, which show a loss from day to day, due probably to volatilization of nicotin. After the first twenty-four hours the loss was inconsiderable (about 0.5 per cent for each twenty-four hours) considering the errors incident to titration.

Notwithstanding this loss, the results after standing with the strong soda twenty-four hours are somewhat higher than by the Kissling method. This may be due in part to incomplete removal of ammonia, but I am convinced that this excess consists chiefly of pyridin bases or some other difficultly volatile alkaline substances.

Perhaps the experiments which I have cited to show that ammonia is completely driven off from a solution by treatment with strong soda for twenty-four hours may not prove the point. Ammonia water and soda mix perfectly, whereas a solution of nicotin, when treated with soda, at once separates into an oily nicotin layer, which floats, and an aqueous solution. This separation may prevent perfect contact of the soda with the ammoniacal solution, or the nicotin layer may interfere with the volatilization of the alkali. I think, however, that the results indicate that the ammonia is removed and that the whole trouble with both the Kissling method and the method without extraction is that pyridin bases are present in the nicotin distillate, the quantity being greater in the latter case. As the pyridin bases are most of them rank poisons, it may be urged with reason that the value of tobacco extracts does not depend entirely on the nicotin, and therefore a conventional method should be employed which, like the above, includes with the nicotin the largest amount of other poisonous bases.

The results of all the w rk that has been done show very clearly that more accurate methods should be secured for the determination of nicotin in tobacco. The tobacco dips are of very great importance and usefulness in the treatment of sheep for scab. When these tobacco dips are used for this purpose it is important to note what percentage of nicotin is present in the solution used in order to be sure that there is sufficient quantity to have the desired effect, and at the same time not too large an amount to injure the animal. I would suggest, therefore, that a committee or reporter be appointed by the association in order to see if some method can be devised which shall be considered as thoroughly standard and reliable as methods in other lines of work which have been adopted and approved by the Association of Official Agricultural Chemists.

At the conclusion of the reading of the paper it was voted that a referee and associate referee be appointed on insecticides, to which the matters left in doubt as indicated in the above paper should be referred.

The president then called upon Mr. E. W. Allen for the report of the abstract committee, which was presented, as follows:

REPORT OF THE COMMITTEE ON ABSTRACTS.

Mr. President and Members of the Association: Your committee has the honor to report that its work during the past year has been conducted on the same general plan as in previous years. Immediately after the appointment of the members of the committee correspondence was instituted to ascertain what journals were accessible to each member. Following this an assignment of the journals was made to the individual members, with a view to distributing the work as evenly as circumstances would permit. The following is a copy of the letter sent to each member of the committee, indicating the assignment of journals and making suggestions for the work:

November 23, 1897.

———— ————,

————— ————.

Dear Sir: The following is a list of the periodicals assigned to the different members of the abstract committee for 1897–98:

E. W. Allen: Forsch. Ber. Lebensmtl.; Ztschr. Nahr. Untersuch. u. Hyg.; abstracts in Analyst, Chem. Centbl., and Chem. Ztg. Repert.

J. T. Anderson: Chem. Ztg.; Jour. prakt. Chem.; Amer. Jour. Sci.

W. H. Beal: Bul. Soc. Chim. Paris; Compt. Rend. Acad. Sci. Paris; Forsch. Geb. Agr. Phys.; Ztschr. anorgan. Chem.; abstracts in Jour. Chem. Soc. [London].

B. H. Hite: Chem. News; Bul. Assn. Belge. Chim.; Jour. Pharm. et Chim.

J. B. Lindsey: Ztschr. angew. Chem.; Ztschr. physiol. Chem.; Jour. Landw.

L. H. Merrill: Ztschr. analyt. Chem.; Jour. Chem. Soc. [London]; Analyst.

A. A. Persons: Jour. Amer. Chem. Soc.; Rev. Chim. Analyt. et Appl.

J. P. Street: Amer. Chem. Jour.; Jour. Soc. Chem. Ind.; Landw. Vers. Stat.; Ber. deut. chem. Gesell.

F. W. Woll: Scandinavian periodicals; Milch Ztg.; Neue Ztschr. Rübenz. Ind.

It will be understood that this committee undertakes to abstract only articles relating to methods and apparatus for agricultural analysis. In this field it is very desirable that everything contained in the journals assigned should be either abstracted or listed by title, with a few words of explanation, according to the importance of the article. In case you do not desire to become responsible for all of the journals assigned to you will you please notify the chairman at once, so that other arrangements may be made. The Office of Experiment Stations desires to make the review of methods of agricultural analysis as complete as possible, and to that end will, as heretofore, supplement the work of the abstract committee.

It has been thought best to have the work of the new committee begin with journals issued on or after October 15, 1897. For the form of statement of the abstract reference is made to Experiment Station Record. In giving the reference please state the name of the periodical, using the abbreviations given above, the volume number in figures, the year in parentheses, the number within the volume (No., Heft, Fasc., etc.), and the pages (beginning and end of the article) with the number of figures. The length of the abstract should be governed by the importance and value of the article, so far as practicable. Articles not deemed worthy of abstracting should be listed by title; usually a few words of explanation of the nature of the article may be added with advantage. Abstract only the original papers in the journals assigned, as the abstracts, where the journals contain such, are provided for. Please take pains to give full references and to write all names and technical terms plainly. The abstracts should be signed by the abstractor.

Both on account of the increased value and the convenience in publishing the abstracts, it is desirable that the work should be kept well up to date and not allowed to accumulate. There is necessarily considerable delay in publishing the Experiment Station Record, as is the case with most Government publications.

I send you herewith a supply of franked envelopes to be used in sending in material. Others can be procured as needed.

Very respectfully, yours, E. W. Allen, *Chairman.*

The publication of the abstracts made by the new committee began in Experiment Station Record, Vol. IX, No. 4, and has continued quite regularly through the year up to Vol. X, No. 3, which is now in press. The printed work of the committee occupies about 60 pages. The effort has been to confine the work more strictly than heretofore to methods and apparatus for agricultural analysis in a broad sense.

Two of the members of the committee were obliged to withdraw from the work early in the year before scarcely anything had been done, and in some individual cases it has not been possible to keep the abstracting up to date. In such cases the attempt has been made to so supplement the work in the Office of Experiment Stations, that the abstracting of the journals would not be allowed to fall behind to any considerable extent. It is believed that in most lines the field has been covered in a general way and that the work is quite well up to date.

E. W. Allen,
J. T. Anderson,
W. H. Beal,
B. H. Hite,
J. B. Lindsey,
L. H. Merrill,
A. A. Persons,
J. P. Street,
F. W. Woll,
Committee.

The report of the committee on food standards was next called for, and read by Mr. William Frear, as follows:

REPORT OF THE COMMITTEE ON FOOD STANDARDS.

STANDARDS.

At the session of 1897 this association appointed Messrs. H. W. Wiley, E. H. Jenkins, M. A. Scovell, H. A. Weber, and William Frear as a committee on food standards to consider the practicability of compiling, for the use of the increasing proportion of the association's membership charged with the duty of official food control, all accessible and trustworthy chemical and physical data as to the composition of foods and drinks for man and domestic animals offered for sale on the American market. This committee was authorized to initiate, without further action by the association, any such work as it might deem practicable and useful.

Your committee presents herewith, as a report of progress, the following statements:

Your committee, as the result of personal conference and correspondence, was unanimously of the opinion that European standards were not universally applicable to American food stuffs, and that the chemical methods employed by food analysts in other countries to determine the purity or the character of adulteration of foods were not always to be relied upon by American analysts, for the reason that in this country processes of food manufacture and adulteration are in vogue different, in many instances, from those practiced elsewhere.

The desirability of the compilation of authoritative data which might serve to the food analyst and manufacturer the same purpose as that portion of the United States Pharmacopœia relating to the composition and purity of drugs serves to the pharmacist was recognized, and it was deemed important that such compilation should be begun without further delay. The attempt to formulate a set of standards, except upon the basis of such a compilation, is regarded as premature and likely to cause confusion, though already accessible data make possible, for most of the important foods, the early formulation of a set of provisional standards.

Your committee, therefore, decided to organize at once the work of compiling from the best selected sources the following data:

1. The maximum, minimum, and average proportion of the most characteristic components in normal food substances, which in kind and proportion best serve to show the source of the product and which, in samples of unknown origin, best show whether they are pure or have been in any way tampered with.

2. The variations in these proportions due to differences in origin or treatment, as place of growth, stage of development, method of manufacture, etc.

3. Data showing the maxima for injurious constituents that occur naturally in food products, and indicating the maximum limits beyond which such substances, introduced incidentally during manufacture or to serve as preservatives, coloring agents, etc., can not be allowed to pass without menace to the health of the consumer.

For the better prosecution of this work the cooperation of the leading food chemists of America and foreign countries was invited.

The preliminary organization of the work consisted in the appointment of three subcommittees.

(A) To formulate rules to govern the selection of data, fix upon forms for their arrangement, and establish regulations needful for the bibliographic completeness of the work, so that the critical character of the work may be maintained, that it may itself invite intelligent criticism, and that the interpretation of data may be simplified, so far as possible. Also, to classify the subject-matter so that it may be satisfactorily subdivided and assigned to several editors and subeditors. Subcommittee: Messrs. Frear, Scovell, and Wiley.

(B) To prepare for the use of the committee a complete directory of official food chemists—national, State, and municipal, American and foreign. Subcommittee: Messrs. Weber, Jenkins, and Wiley.

(C) To prepare an analytical digest of existing food legislation: Messrs. Jenkins, Scovell, and Wiley.

Subcommittee A having recommended a classification of the entire subject-matter of foods and drinks, referees or editors were appointed, as follows, to work upon the compilation of analytical data and recommend, after consultation with all trade interests directly affected, suitable definitions and standards for the several food substances:

I. Animal Products:

A. *Meat*—
(a) Raw, canned, pickled, salted, smoked, sausage.
(b) Meat extracts, peptones, etc.
(c) Lard.
(d) Eggs.

Referee, Dr. Charles D. Woods, director, Maine Agricultural Experiment Station, Orono, Me.

B. *Milk and its products*—
(a) Milk, skim milk, cream, butter, buttermilk, cheese, whey, koumiss, condensed, sterilized, pasteurized, conserved milk.
(b) Ice cream and ices.
(c) Infant foods.
(d) Butter substitutes and cheese substitutes.

Referee, Dr. L. L. Van Slyke, chemist, New York Agricultural Experiment Station, Geneva, N. Y.

II. Vegetable Products:

C. *Grains and their products*—
(a) Cereals and legumes. Flour. Prepared meals and starches.
(b) Bread and pastry, puddings, etc.
(c) Cattle foods.

Referee, Dr J. B. Lindsey, chemist, Massachusetts Agricultural Experiment Station, Amherst, Mass.

D. *Fruit and vegetables*—Dried, canned, preserved, fruit butters, jams, etc.

Referee, Prof. H. A. Weber, chemist to Dairy and Food Commissioner, Columbus, Ohio.

E. *Sugar and related substances*—
(a) Sugar, molasses, sirups—cane, sorghum, maple, refinery, glucose, etc.—solid glucose.
(b) Confectionery.
(c) Honey.
(d) Other sweetening agents.

Referee, Prof. M. A. Scovell, director, Kentucky Agricultural Experiment Station, Lexington, Ky.

F. *Condiments (except vinegar)*—
(a) Spices.
(b) Fruit extracts and artificial flavors.
(c) Salad oils.
(d) Salt.

Referee, Dr. A. L. Winton, chemist, Connecticut Agricultural Experiment Station, New Haven, Conn.

G. *Drinks and vinegar*—
(a) Infusions—tea, coffee, cocoa, chocolate, cereals.
(b) Fruit juices, fresh and sweetened. Fermented fruit juices—cider, perry, wine.
(c) Vinegar.
(d) Mead, root beer, etc.
(e) Malted liquors.
(f) Spirituous liquors.
(g) Carbonated and other similar beverages.

Referee, Dr. Charles A. Crampton, chemist, Bureau of Internal Revenue, Washington, D. C.

H. *Substances artificially introduced with possible injury to health*—
(a) Preservatives.
(b) Coloring matters.
(c) Metals and metallic salts.
(d) Other injurious substances

Referee, Prof. H. A. Huston, State chemist of Indiana, Lafayette, Ind.

Subcommittee also submitted to the several editors a statement relative to the selection of data, the form in which they should be recorded with highest value for reference and critical examination, and to the principles of formulation of standards approved by the committee. Full consultation with all trade interests concerned was especially requested in the framing of definitions and formulation of standards.

Owing to the magnitude of this work it is not possible as yet for the editors to announce the full organization of their respective divisions of the work.

The committee may, however, announce that it is already in receipt of a most valuable compilation of existing data relative to the composition of American and European salad oils, by Prof. G. E. Colby, of the University of California; that Prof. F. W. Woll, of the University of Wisconsin, is now engaged upon an extensive study of table and dairy salts upon which, together with previously existing data, he hopes to offer recommendations; also, that Mr. A. L. Winton, owing to the dearth of American analyses of spices imported into this country, has, through the courtesy of a number of New York and Boston importers, sampled in original package more than 100 lots of the various spice materials and is now engaged upon their chemical and microscopic examination. The stimulation and suggestion of new and important lines of food investigation indeed promise to be of the most important results of the labors of the committee.

It is with pleasure that the committee notes the cordial cooperation of many of the leading manufacturers of the country in assisting the several referees in their laborious task.

In closing, your attention is called to the action of the National Pure Food and Drug Congress, that met in this city on March 3 to 5 last, composed of delegates represesenting most of the food trade, health and food control interests of America. This congress distinctly approved the enactment of a law establishing a commission to be composed of representatives appointed by the Association of Official Agricultural Chemists, together with others appointed by the American Chemical Society and of physicians, to be appointed by the President of the United States from the Surgeon-General's bureaus of the War and Navy Departments; which commission shall, upon request from the Secretary of Agriculture, and after due hearing of all trade interests, recommend to the Secretary of Agriculture, for use in United States courts, standards for such food substances as he may name.

In view of the general demand for the prosecution of the work intrusted to your committee and of the laborious nature of the tasks assumed by the several editors and subeditors, the committee bespeaks for each of them the most cordial cooperation of every member of the association.

H. W. Wiley, *Chairman.*
H. A. Weber.
M. A. Scovell.
E. H. Jenkins.
William Frear.

The report of the committee on food standards was adopted, and it was voted that the committee be continued for the purpose of completing the work.

Mr. H. J. Wheeler then read the following report of the committee on uniform methods of fertilizer control:

REPORT OF COMMITTEE ON UNIFORM METHODS OF FERTILIZER CONTROL.

STATEMENT OF PRINCIPLES.

(1) All substances containing nitrogen, potash, or phosphoric acid sold, offered, or exposed for sale for manurial purposes, excepting the dung of domestic animals when sold as such, should be subjected to inspection.

(2) Each package of manurial substance as above defined sold, offered, or exposed for sale should bear a printed, legible guaranty.

(3) The number of net pounds in each package should be stated.

(4) Each package should bear the brand, name, or trade-mark, and the name and address of the manufacturer.

(5) The quantities of manurial ingredients should be expressed in percentages.

(6) The guaranteed statements on the packages or forwarded to the inspectors need not be in the form of an affidavit.

(7) Sealed samples of fertilizers offered for sale need not be sent by the manufacturers to the inspectors.

(8) In lots of 5 tons or less samples should be drawn from at least 10 packages, or if less than 10 packages are present all should be sampled; in lots of over 5 tons not less than 20 packages should be sampled.

(9) Duplicate samples should be drawn and sealed in the presence of the party or parties in interest or their representatives, one of the samples to be taken by the collector and the other left with the party whose goods were inspected, subject to the call of the manufacturer.

(10) Total nitrogen should be guaranteed in all cases, and nitrogen in the form of nitrates or of ammonium salts should be guaranteed separately if the manufacturer desires credit therefor.

(11) Total phosphoric acid should be guaranteed in all cases, and soluble reverted and insoluble, separately, if the manufacturer desires credit therefor.

(12) Potassium should be guaranteed as potash (potassium oxid) soluble in water.

(13) When chlorin is present in fertilizers, manufacturers shall guarantee the maximum percentage thereof.

(14) Only statements of the minimum amount of the substances present, except in the case of chlorin, should be made. For example, instead of guaranteeing from 2 to 4 per cent of potash guaranties should read thus: 2 per cent of potash.

(15) It should be specified that the methods of the Association of Official Agricultural Chemists should be used for official work.

(16) An imprisonment clause for violation of fertilizer laws should be omitted.

(17) Manufacturers should not be obliged to secure bondsmen.

(18) Commercial valuations should not be affixed to the published analyses of manurial substances.

(19) A definition of the term "brand" should be inserted wherever a brand tax is assessed.

(20) The guaranty should state the ingredients guaranteed, in the following form and order:

Per cent phosphoric acid soluble in water.
Per cent phosphoric acid reverted.
Per cent phosphoric acid insoluble.
Per cent phosphoric acid total.
Per cent nitrogen in nitrates.
Per cent nitrogen as ammonia.
Per cent nitrogen total.
Per cent potash soluble in water.
Per cent chlorin.

Ingredients not present should be omitted from the guaranty and no other form of statement should be interpolated.

It is further recommended that this committee be continued as a standing committee on fertilizer legislation.

H. W. Wiley, *Chairman.*
B. W. Kilgore.
H. B. McDonnell.
H. A. Huston.
B. B. Ross.

The committee appointed by the Association of American Agricultural Colleges and Experiment Stations to confer with the committee from the Association of Official Agricultural Chemists on uniformity in fertilizer laws, concurs with the committee in recommending the adoption of the above report as a guide which should be followed by the several States in the enactment of fertilizer laws and by the Congress of the United States should a law regulating interstate traffic in commercial fertilizers be enacted. H. J. Wheeler, Chairman; H. P. Armsby, E. H. Jenkins, M. A. Scovell, C. D. Woods, committee.

The thanks of the association were given to the Secretary of Agriculture, the retiring president, the secretary of the association, the Cosmos Club, and President Whitman and Dean Munroe of the Columbia University.

The convention adjourned at 1 p. m., to meet at time and place to be selected by the executive committee.

OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1899.

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Mr. H. W. WILEY, Washington, D. C.

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Mr. B. B. ROSS, Auburn, Ala.
Mr. WM. FREAR, State College, Pa.

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Nitrogen.—Mr. F. S. Shiver, Clemson College, S. C.
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Soils and ash.—Mr. B. L. Hartwell, Kingston, R. I.
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Foods and feeding stuffs.—Mr. Thorn Smith, Moscow, Idaho.
Liquor and food adulteration.—Mr. H. A. Weber, Columbus, Ohio.
Sugar.—Mr. Elton Fulmer, Pullman, Wash.
Tannin.—Mr. Oma Carr, Corry, Pa.
Insecticides.—Mr. E. A. de Schweinitz, Washington, D. C.

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Nitrogen.—Mr. W. R. Perkins, Agricultural College, Miss.
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Soils and ash.—Mr. M. E. Jaffa, Berkeley, Cal.
Dairy products.—Mr. F. W. Woll, Madison, Wis.
Foods and feeding stuffs.—Mr. C. A. Browne, jr., State College, Pa.
Liquor and food adulteration.—Mr. W. D. Bigelow, Washington, D. C.
Sugar.—Mr. G. L. Spencer, Washington, D. C.
Tannin.—Mr. E. J. Haley.
Insecticides.—Mr. L. A. Voorhees, New Brunswick, N. J.

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Mr. J. T. Anderson, Auburn, Ala.
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Mr. M. A. Scovell, Lexington, Ky.
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Mr. Wm. Frear, State College, Pa.

COMMITTEE ON UNIFORM LAWS FOR FERTILIZER CONTROL.

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COMMITTEE ON REWRITING METHODS OF SOIL ANALYSIS.

Mr. M. A. Scovell, Lexington, Ky., chairman.
Mr. A. M. Peter, Lexington, Ky.
Mr. H. W. Wiley, Washington, D. C.

COMMITTEE ON REWRITING BULLETIN NO. 46.

Mr. H. W. Wiley, chairman, with associates to be selected by him.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects of the association shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership; and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members and to have all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote

(3) The officers of the association shall consist of a president, a vice-president, and a secretary, who shall also act as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the president, at the regular annual meeting, a referee and associate referee for each of the subjects to be considered by the association.

It shall be the duty of these referees to prepare and distribute samples and standard reagents to members of the association and others desiring the same; to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) No changes shall be made in the methods of fertilizer analyses, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of fertilizer work to test the proposed changes.

(8) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(9) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(10) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

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